## AMELIORATING SOILS WITH RECYCLED RESIDUAL PAPER PULP TO IMPROVE SOIL PHYSICAL AND CHEMICAL PROPERTIES

by

#### **DAMIANOS DAMIANIDIS**

(Under the Direction of Miguel Cabrera and George Vellidis)

#### **ABSTRACT**

Disposal of recycled residual paper pulp (RRPP) is a serious environmental challenge. In this study, the effects of RRPP were evaluated over two years on a clay loam and a sandy loam amended with 0, 1, 2, and 4% volume/volume (v/v) rates incorporated into the upper 0.15 m of soil. Soil properties measured included bulk density, organic carbon (SOC), pH, electrical conductivity, volumetric water content, total nitrogen and heavy metals. Furthermore, effects of RRPP on leaf chlorophyll and fresh yield of *Brassica oleracea*, *Lactuca sativa*, and *Zea mays var. saccharata* were examined. Results indicate a statistically significant increase in SOC for the 4% v/v pulp application rate in sandy loam during both years and in clay loam during the second year. Application rate affected pH significantly only in the sandy loam on both years. All RRPP application rates resulted in proportional yield increases for most crops in both soils.

INDEX WORDS:

Recycled residual paper pulp, clay loam, sandy loam, soil organic carbon, soil amendment, yield, leaf chlorophyll level, bulk density, total nitrogen, heavy metals, pH, volumetric water content, SPAD units, soil amelioration, paper sludge.

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B.S.A., University of Georgia, 2003

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### DEDICATION

To those who never stopped asking questions.

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#### **CHAPTER 1**

#### INTRODUCTION

Paper production processes generate large amounts of byproducts. The paper pulp industry is the sixth largest producer of industrial pollutants, following the oil, cement, leather, textile, and steel industries (Ali and Sreekrishnan, 2001). The pollutants from the paper pulp industry are classified as gases, effluents, and solid wastes. The last category includes recycled residual paper pulps (RRPP), which are also known as solid clarifiers and paper mill sludges (Levy and Taylor, 2003). Wastewater treatment processes at pulp and paper mills include primary treatment, where primary sludge is produced after neutralizing, screening, and sedimenting the suspended solids (Beauchamp et al., 2002; IFC, 2007). Secondary sludges include wastes from the secondary or biological treatment systems, where wastewater organic content is reduced. De-inking sludges include paper pulp residues derived from the removal of ink from recycled paper. Combined sludges are derived from combined primary and secondary sludges.

Disposal of paper pulp residues is a serious challenge to pulp and paper industry. In the United Kingdom alone, the pulp and paper industry produces 250,000 dry tonnes of sludge per year (Phillips et al., 1997). In Canada, 1.7 million dry tonnes of paper sludges are generated every year (Bostan et al., 2005). The most common method of disposal is landfilling (Levy and Taylor, 2003; Nunes et al., 2008; Phillips et al., 1997). Stringent environmental regulations and increased cost have forced the industry to search for disposal alternatives. Incineration is one

alternative (Vagstad et al., 2001), but the high water content of sludge hampers efficient combustion (Phillips et al., 1997).

Another alternative is to use the byproducts as soil amendments. Depending on the treatment process, sludges are characterized by modest concentrations of N, P, and K (Levy and Taylor, 2003). Applying residual paper pulp on agricultural lands is a promising disposal option because it improves soil physical properties, recycles carbon to the soil, and is cost effective. Ameliorating soils with pulp mill solids and de-inking sludge increases soil organic matter content, improves soil water holding capacity, increases macro-aggregate formation, stimulates bacterial enzyme activity, and increases pH level of acidic soils (Levy and Taylor, 2003; Nunes et al., 2008). Furthermore, paper pulp sludges are rich in cellulose and lignin fibers for organic matter formation, yet contain low levels of organic pollutants (Levy and Taylor, 2003; Hojamberdiev et al., 2007). In addition, paper sludges contain inorganic fillers and coating materials (i.e. kaolinite, limestone, and talk).

Mediterranean soils tend to be low in organic matter content due to climatic conditions, which are characterized by a prolonged hot and dry summer, followed by heavy precipitation during the fall (Nunes et al., 2008). Additionally, in Greece not optimal management practices in the past, other natural and anthropogenic causes, resulted in soils having low levels of organic matter (Koukoulakis et al., 2000). More specifically, average soil organic matter % in soils of North Greece is approximately 1.15% and following a trend of 0.8% annual decrease of the average value. Therefore, low soil organic matter is considered as one of the major problems in soil productivity of Greek soils. The main soil properties which affect crop growth and yield and require improvement are soil organic carbon, pH, cation exchange capacity, and water holding

capacity. Spreading residual paper pulp on those lands may improve these soil conditions (Phillips et al., 1997) and may increase crop yield (Vagstad et al., 2001).

MEL Macedonian Paper Mills S.A., located at Kato Gefyra in Greece, is the Greek sole manufacturer of cardboard using recovered raw materials, and generates approximately 20 dry tonnes of RRPP per day (S. Karapatakis, personal communication). This research evaluated the use of RRPP in dry and pulverized form as a soil conditioner.

The objective of this study is to determine, during an initial two year period, the effect from application of various rates of RRPP on: a) main chemical and physical properties of two soils (a clay loam and a sandy loam) which impact crop productivity, and b) productivity characteristics (yield and relative leaf chlorophyll level) of *Brassica oleracea* L (cabbage), *Lactuca sativa* L (lettuce). and *Zea mays* var. L. *saccharata* (sweet corn) grown in rotation and under low input management approach (Low Input Sustainable Agriculture-LISA) on these soils.

#### **CHAPTER 2**

#### LITERATURE REVIEW

#### Paper and pulp production

Production of paper and board globally reached 367 million tonnes in 2005 (Mies et al., 2006). Pulp production alone for the same year increased by 0.4% to 189 million tonnes. In 2005, the United States led the world by producing 41.4, 49.7 and 59 million tonnes of paper, board, and pulp, respectively. Preliminary statistics for 2006 based on annualized production rates predict similar production outputs for the United States. China, which was in second place for 2005, produced 56 million tonnes of paper and board. After China, the top paper and board-producing countries were Japan (30.9 million t), Canada (21.7 million t) and Germany (19.5 million t). In 2009, world paper and board production was estimated at 380 million tonnes (PWI, 2009). In the case of Greece, there are no reliable data.

Europe plays a significant role in paper production by having the second largest industry in the world after Asia (EC, 2001a; Mies et al., 2006). Paper and board production in Europe surpassed North America's production for the first time in 2002, when more than 101.3 million tonnes were produced. The last available data (2005) reveal Asian countries contribute more than 1/3 of the world's board and paper total production. Even so, North America's pulp industry remains the world's leader in the pulp production.

Paper-making involves four main stages: a) raw material preparation, b) pulp manufacturing, c) pulp bleaching, and d) paper manufacturing (MIGA, N/A). Pulp and paper mills are either separated or integrated. Manufacturing paper and pulp requires raw materials

containing cellulose fibers that are derived from wood, recycled paper, and agricultural residues (i.e., cotton, hemp, straw) (IFC, 2007; MIGA, N/A).

If wood is the source of cellulose for pulp and paper production, then the raw material preparation will include debarking of logs and wood chipping (IFC, 2007; MIGA, N/A). Pulp manufacturing efficiency and pulp quality requires uniform chip size (20 mm long and 4 mm thick). Nonwood fibers undergo specific handling related to their composition to minimize fiber degradation and to optimize pulp processing.

In plant tissue, cellulose and hemicelluloses fibers are bound together by lignin (IFC, 2007). During pulping process the cellulosic rich raw material is broken down to individual fibers. Pulp manufacturing is separated into two broad categories: chemical pulping and mechanical pulping. Pulping methods involve solely mechanical or solely chemical pulping procedures or are combined together in a procedure known as chemi-mechanical pulping. Mechanical pulping uses mechanical means such as disk abrasion to separate fibers. A modification of the mechanical pulping are the thermo-mechanical and the chemithermomechanical pulping processes (IFC, 2007; MIGA, N/A). Thermo-mechanical pulps processes combine steam chip pretreatment with mechanical operations that reduce chip sizes. Newsprint paper is produced from thermomechanical pulps. In chemi-thermomechanical and chemi-mechanical pulping processes, sodium sulfite, carbonate, or hydroxide are needed to soften the raw material. Chemical pulp results from digesting the raw material either with sulfate (Kraft process) or with sulfite. High strength paper and boards are made from Kraft pulp. Furthermore, producing pulp from recycled paper is common procedure. Nowadays, recycled fiber accounts for 50% of the raw fiber material utilized in paper-making (IFC, 2007).

Any process that alters pulp characteristics and increases pulp brightness is called bleaching (IFC, 2007). Generally bleaching gives papers that are whiter, softer, brighter, and have larger absorbent capacities compared to papers produced from unbleached pulps. Reducing lignin content of the pulp is the focus of bleaching processes. Pulp generated from mechanical processes is high in lignin content (MIGA, N/A). If the brightness of the final product is not an issue (i.e., newsprint paper), then bleaching is unnecessary. However, for printing, copying, and some packaging paper grades, bleaching is necessary. Bleaching agents for mechanical pulps are peroxides and hydrosulfites. For chemical pulps, the bleaching process removes the small lignin fraction resulting from cooking (Kraft or sulfite processes). Transforming lignin into an alkali soluble form is achieved by employing oxygen, hydrogen peroxide, ozone, peracetic acid, sodium hypochlorite, chlorine dioxide, and chlorine. An alkaline solution, such as sodium hydroxide, is necessary to extract the alkali soluble lignin from the pulp.

In modern mills, oxygen is a common bleaching agent (MIGA, N/A). From environmental perspective, elemental chlorine as bleaching agent is not recommended because soluble organic compounds from the bleaching stage are chlorinated. Some of the chlorinated organic compounds are extremely toxic (i.e., dioxins, furans, and chlorinated phenols). Therefore, the trend is to move towards elemental chlorine free (ECF) bleaching processes, and if possible, toward total chlorine free (TCF) bleaching processes.

A sheet of paper or cardboard results from the deposition of cellulose fibers and fillers from liquid suspension on a moving device (EC, 2001a). Gravity, vacuum chambers, and vacuum rolls are used to remove excess water from the slurry (IFC, 2007). Pressing the continuous sheet in a series of cylinders removes more water and compresses the fibers. At the

drying section, fibers bond together as steam-heated cylinders compress the sheets further. Finally, a coating is applied to improve gloss, color, printing detail, and brilliance.

#### Waste

Significant environmental impacts result from the operation of pulp and paper industry (IFC, 2007; MIGA, N/A; Hojamberdiev et al., 2007). Impacts relate to generation of wastewaters, air emissions, solid wastes, and noises. Estimates of wastewater discharges from pulp and paper manufacturing processes range from 10 to 250 m<sup>3</sup> t<sup>-1</sup> of product (IFC, 2007). For the pulp mill, the term "product" is 90% air-dried pulp; for paper and board industry it is the weight of sold paper. Gaseous emissions vary from mill to mill because production procedures may differ at stage of production.

Solid wastes from paper fabrication include bark from debarking processes of wood, inorganic sludges from chemical recovery (e.g., green liquor, lime sludge), and plastics separated from paper during the recycling process in the recovered fiber plants. In the past, wastewaters were discharged directly to waterbodies and contributed to environmental degradation (Oral et al., 2005). This problem was solved by introduction of wastewater treatment facilities as a standard method for treating effluents. The downside of this introduction was the generation of large amounts of biosolids.

Biosolids are classified as primary or secondary sludges depending on whether they are the product of a primary or secondary wastewater treatment process, respectively (IFC, 2007; MIGA, N/A). Production procedures in a pulp mill account for material losses of 3 to 4% because of inefficient solid/liquid separation at the successive manufacturing stages (Mahmood and Elliott, 2006). Alternatively, in a paper plant, material losses are in the order of 15 to 30%. The lost material is captured by gravity sedimentation at the primary clarifier. Those primary

treatment residues consist of cellulose and lignin fibers, fines, and fillers (i.e., calcium carbonate or clay). According the MIGA (N/A), the pulp and paper industry generates 50 to 150 kg of biosolids per ton of air-dried pulp. Any increase in paper production with broader introduction of secondary or activated sludge process for wastewater treatment amplifies biosolids management problems because more total waste solids need disposal.

The nutritive value of paper pulp sludges varies among various sludges, and their constituents depending on the specific procedures followed during the paper- and pulp-making processes (Tucker and Douglas, 2006). Compared to municipal sewage sludges, residual paper pulps contain lower levels of N and P while, concentrations of Ca and Mg are much higher. Generally, primary sludges are lower in nutrients concentrations compared to secondary sludges. For example, secondary or biological sludges tend to have higher concentration of N. As a result, the C/N ratios of primary paper sludges are usually high (>100:1), but this again varies from mill to mill. Recycled residual paper pulps are solids of high organic content. De-inking sludges are lower in organic constituents compared to paper sludges derived from other paper and pulp processes because they contain high concentration of ash (30 to 80%).

Nowadays, heavy metal concentrations of de-inked sludges are lower than the established thresholds of environmental concern. In some de-inked sludges, though, Cu concentration are elevated because of the blue and green phthalocyanine pigments used in several color ink formulations. Average Cu concentrations of paper pulp sludges range from 2 to 349 mg kg<sup>-1</sup> (EC, 2001b; Tucker and Douglas, 2006). These concentrations are similar with concentrations of other byproducts such as pig slurry (180 to 574 mg kg<sup>-1</sup>), pig manure (mean=346 mg kg<sup>-1</sup>), cattle manure (15 to 75 mg kg<sup>-1</sup>) and cattle slurry (31 to 70 mg kg<sup>-1</sup>) (EC, 2001b). In contrast, Cu concentrations in sewage sludges are often in excess of 1000 mg kg<sup>-1</sup> (Tucker and Douglas,

2006). Other heavy metals present in de-inked sludges are Ba and Cr, derived from the coating materials and fillers detached during the de-inking process. Additionally, pulp and paper sludges may contain Zn (1.3 to 330 mg kg<sup>-1</sup>), (<1 to 83 mg kg<sup>-1</sup>), Cr (<1 to 44 mg kg<sup>-1</sup>), Ni (<1 to 32 mg  $kg^{-1}$ ), Hg (<1, to 1.4 mg  $kg^{-1}$ ), Cd (0 to 4 mg  $kg^{-1}$ ), and As (<8 mg  $kg^{-1}$ ) (EC, 2001b). Polychlorinated biphenyls (PCBs), dioxins, furans, and halogenated hydrocarbons are usually not detected in paper pulp sludges or are present in extremely low levels (Tucker and Douglas, 2006). When *Pinus radiata* (and other conifers, as well) is the predominant raw material for pulp production the effluents contain large concentrations of resin acids (Fraser et al., 2009). Aerated stabilization of effluents (secondary treatment) lead to biotransformation of resin acids to resin acids neutrals (i.e. retene), both involved in aquatic toxicity derived from paper mill effluents. Concentrations of polycyclic aromatic hydrocarbons (including retene) may have larger concentrations in the solid wastes derived from pulp and paper mill effluents. Nevertheless, Fraser et al. (2009) reported that land application of pulp and paper solids was not lethal to the soil invertebrates and plants grown on the experimental plots, though there were reproductive effects on enhytraeid worms. Furthermore, pathogens in paper pulp sludges are not an issue of environmental concern (Tucker and Douglas, 2006).

Dioxins are a group of toxic chemicals which have long been associated with the pulp and paper industry. The dioxins family includes a large group of chemical compounds that do not readily degrade in the environment (NRC, 2006). Rather, dioxins persist in the environment and therefore accumulate in trace amounts in animal tissues. The most toxic chemical compound among dioxins is 2, 3, 7, 8-tetrachlorodibenzo-p-dioxin or TCDD (Colborn et al., 1997). Many dioxins and dioxin-like compounds share TCDD's toxic characteristics (NRC, 2006), and therefore TCDD is only the tip of the iceberg.

Nowadays, dioxins are ubiquitous in the environment and are detected in air, soil, and water bodies (Thacker et al., 2007). In contrast to other hormone disrupting chemicals, dioxins are not formulated intentionally (Colborn et al., 1997). Although, dioxins are released during natural processes (volcanic eruptions, and forest fires), it is mostly human activities which are responsible for dioxin formation. Incinerating chlorine-containing wastes, manufacturing certain chlorine-containing chemicals (i.e., pesticides, wood preservatives etc.), burning fossil fuels, and bleaching paper pulp with chlorine results in dioxin formation (Colborn et al., 1997; NRC, 2006; Thacker et al., 2007).

There is sufficient evidence to link exposure to dioxins to human health problems. Exposure to high levels of 2, 3, 7, 8-tetrachlorodibenzo-p-dioxin results in chloracne, a severe skin disease (Colborn et al., 1997). Also, dioxin exposure is linked with birth and developmental defects, learning disabilities, increased risk of diabetes, tumor promotion, decreased fertility, reduced sperm counts, suppressed immune systems, and cancer (Colborn et al., 1997; NRCM, N/A)

Substituting ClO<sub>2</sub> for elemental chlorine as bleaching agent is the modern trend in paper and pulp industry because it results in lower levels of dioxin production. In state of the art pulp mills, elemental chlorine is no longer utilized as a bleaching agent. As a result, in mills were ClO<sub>2</sub> was the bleaching agent 2, 3, 7, 8-tetrachlorodibenzo-p-dioxin ranged from below detectable limits to 0.12 ng L<sup>-1</sup> (Thacker et al., 2007). Dioxin generation is eliminated completely when total chlorine free bleaching is applied (Karapatakis, 2009).

#### **Waste management alternatives**

Pulp and paper wastes can be disposed of several main ways (Curnoe et al., 2006): i.e. landfilling, incineration, and land application. Landfilling remains the primary disposal method

(Nunes et al., 2008). Although convenient, potential environmental hazards because of leaching and ground water contamination, along with increased public opposition and excessive cost, provide good grounds for reconsidering landfilling (Mahmood and Elliott, 2006). Furthermore, the reduction in available landfill space and stricter regulation make landfilling more difficult to implement compared to the past.

Incineration provides an alternative for the handling of sludge resulting from the wastewater treatment process of pulp and paper plant (Oral et al., 2005). Additionally, incineration of pulp and paper sludges recovers energy (Tucker and Douglas, 2006).

Nevertheless, incineration has drawbacks as well, including increased fuel prices, high capital costs, and air pollution concerns (Nunes et al., 2008). Furthermore, the high water content of sludge hampers efficient combustion (Nunes et al., 2008; Phillips et al., 1997).

A third way to dispose of pulp and paper wastes is land application. Using industrial organic byproducts as soil conditioners in farmlands is a means to convert a waste product to a value-added resource (Foley and Cooperband, 2002). Direct land application of paper pulp sludge (primary, secondary, or de-inked) is an attractive alternative for disposing of recycled residual paper pulp (Tucker and Douglas, 2006). On farmlands, the RRPP application rate ranges from 15 to 30 t/ha (EC, 2001b; Price and Voroney, 2007). Because recycled residual paper pulps are low in inorganic nutrients, the application rate-determining factors are the CaCO<sub>3</sub> content and/or the level of N deficiency induced if sludges with high C/N ratio are applied. Practically, heavy metals are usually not a limiting factor because of their low concentration levels.

Nevertheless, lack of long-term research on the application of residual paper pulp on agricultural land and several public safety concerns are two factors that limit the widespread adoption of this disposal method (Price and Voroney, 2007).

#### Effects of paper pulp amendments on soil physical and chemical properties

According to Price and Voroney (2007), ameliorating four different soils with deinked paper pulp residues high in C/N ratio reduced soil bulk density, increased wet aggregate stability, and increased surface water infiltration. Furthermore, recycling C into the soil by ameliorating soils with organic by-products is achievable (Foley and Cooperband, 2002). Applying residual paper pulp on coarse loam and loamy sand increases total C (Foley and Cooperband, 2002; Price and Voroney, 2007). Treating low total soil C (loamy sand) with paper mill residues, composted paper mill residues, and paper mill residues composted with bark resulted in large increases in total soil C (Foley and Cooperband, 2002). One year after the application, the treated plots maintained 60 to 115 % more total soil C than unamended (control) plots. Ameliorating two soils (a Cambric Arenosol and Cromic Cambisol) under greenhouse conditions with secondary paper sludge at application rates of 40, 80, and 120 t ha<sup>-1</sup> increased soil organic content (Nunes et al., 2008) even though only the highest application rate (120 t ha<sup>-1</sup>) showed a statistically significant increase in soil organic carbon. Applying de-inked paper fiber to Dystric Gleysols for 3 or more successive years had a site-specific effect on soil organic matter content. While in some sites, no significant increase in soil organic matter was apparent even after successive residual paper pulp applications, in others, soil organic matter content increased (Tandy et al., 2008).

In addition, amended soils with high C/N ratio de-inked paper sludge did not affect soil total N after 2 years (Price and Voroney, 2007). A decrease in total soil N was observed only in a silt loam as a result of increasing rates of paper sludge treatments (50, 100, and 150 Mg ha<sup>-1</sup>). Furthermore, increasing rates and successive applications of secondary sludge on a sandy loam raised NO<sub>3</sub>-N concentrations at the top 15 cm. Nevertheless, up to 45 cm depth NO<sub>3</sub>-N

concentration was constantly below 10 mg kg<sup>-1</sup> (Curnoe et al., 2006). Therefore, the leaching risk for NO<sub>3</sub>-N following residual paper pulp application to the field was minimal.

A decrease in soil bulk density was observed following an increase in application rates of the residual paper pulp (Price and Voroney, 2007). In the coarse sandy loam, a significant decrease in bulk density with increased paper sludge rates was observed in all the three years of the study. In the clay loam, a significant decrease in soil bulk density with residual paper pulp application rates was shown only during the third year of the study.

Furthermore, boardmill sludge is a promising material when applied under conservation tillage practices (Muukkonen et al., 2009), though more research is needed to additionally evaluate the effects of this material on soil microorganisms and nutrient immobilization. The material induced increase in Ca and EC of the surface soil. Also, plant available P increased with increasing pH, while increase in Ca concentration in the soil solution suppressed P concentration, and therefore reduced P losses were observed. Rotenberg et al. (2009) studied the effects of paper mill residual materials on various microbiological and physiochemical soil properties on suppressing soil born disease. Their results provided evidence that bean root rot disease suppression induced by application of paper mill residues into the soil. Consecutive additions of the organic amendment as fresh paper mill residues and composted paper mill residues altered significantly the rhizobacterial communities when compared to the non-amended soils. Furthermore, soil bacterial community composition was significantly influenced from the different types of organic amendments applied.

Application of de-inked paper sludge on four different soil textural classes (silt loam, coarse sandy loam, loam, and clay loam) did not affect pH and electrical conductivity in most of the plots planted with corn at the end of the second year (Price and Voroney, 2007). In contrast,

ameliorating a Cambic Arenosol with residual paper pulp resulted in significant increase in soil pH (Vasconcelos and Cabral, 1993). More specifically, application rates of 30 t ha<sup>-1</sup> increased soil pH by 1 unit compared to the unamended soil. Generally, application rates ranging from 50 to 130 t ha<sup>-1</sup> resulted in pH increase from 1.15 to 2.60 pH units, respectively. These observations suggest that residual paper pulp has significant liming capabilities. Therefore, replacing agricultural lime with paper pulp sludge as liming agent is feasible. In addition, Nunes et al. (2008), reported a significant increase in soil pH by applying secondary paper sludge to two Mediterranean soils (a Cambic Arenosol with an initial pH = 6.40, and a Cromic Cambisol with an initial pH = 5.10). After applying secondary paper pulp to the Cambic Arenosol at rates ranging from 40 to 120 t ha<sup>-1</sup>, the pH increased from 0.97 to 1.20 pH units, respectively. For the Cromic Cambisol, the same application rates (40 and 120 t ha<sup>-1</sup>) increased the pH by 1.60 to 2.23 pH units. Amending Dystric Gleysols with deinking paper pulp raised soil pH in all fields (Tandy et al., 2008). Applying boardmill sludge to Vertic Cambisol in south-western Finland resulted in an average pH increase by 0.3 units in the top 0.1 m soil layer (Muukkonen et al., 2009). Those results are consistent with other studies (Nunes et al., 2008; Vasconcelos and Cabral, 1993), and suggest potential benefits to farmers for fields where acidification is a problem. Filiatraut et al. (2006) investigated in greenhouse experiments the effects of de-inking paper sludge as an organic soil amendment on growth of aspen (*Populus deltoides*) and alder (Alnus crispa) reported that a onetime heavy application rate was effective for soil restoration purposes.

Ameliorating four different soils with residual paper pulp for two years yielded varied soil heavy metal concentrations (Price and Voroney, 2007). In a silty loam, concentrations of As and Cr declined as paper sludge application increased. Similarly, amending a coarse loamy sand

with the same residual paper pulp decreased in Cr and Se concentrations as the rates of application increased. In Cambic Arenosols, heavy metal contamination is not expected as long as residual paper sludge application rates do not exceed 70 to 80 t ha<sup>-1</sup> (Vasconcelos and Cabral, 1993).

Paper pulp biosolids significantly affect aggregate stability after 3 year of application (Price and Voroney, 2007). More specifically, adding paper pulp sludges at a rate of 150 Mg ha<sup>-1</sup> to three different soil textural classes (silt loam, coarse sandy loam, and loam) increase significantly the number of water stable aggregates belonging to size classes 1 to 2, and 2 to 4 mm. This effect was not observed on the first year of application. Also, Nemati et al. (2000a) reported an increase in aggregate stability of soils treated with de-inked paper sludges. The paper sludge application mainly increased the aggregate stability of pores belonging to the 1 to 4 mm size class.

Application of paper pulp residues on soils benefits plant nutrition and tree growth (Bostan et al., 2005). Ameliorating a sandy loam for 5 years with secondary paper sludge at rates of 15 and 25 Mg ha<sup>-1</sup> of dry matter increased corn grain yield (1997-2000) from 2360 to 2908 kg ha<sup>-1</sup> (Curnoe et al., 2006). Yields produced from the two different applications of secondary paper sludge (15 and 25 Mg ha<sup>-1</sup>) were statistically equivalent. Combining dry secondary paper sludge (15 Mg ha<sup>-1</sup>) with 75 kg ha<sup>-1</sup> of NH<sub>4</sub>NO<sub>3</sub>-N consistently produced the highest grain yield. However, grain yield was not significantly different from the treatment where the sludge (15 Mg ha<sup>-1</sup>) was applied with no additional N in four out of five experimental seasons. Applying secondary sludge or NH<sub>4</sub>NO<sub>3</sub>-N to a sandy loam increased total available N to corn plants grown on the amended plots.

Application of paper sludge on excess rates may have negative impacts on receiving waters and plant productivity. Composting of residual paper pulp with high C/N ratio requires additional N to optimize compost processes (Jackson and Line, 1997). Potential leaching of inorganic nutrients and contamination of ground water may result because of poor compost pile management, even though composting reduces the volume and the water content of the residual paper pulp. Furthermore, stabilizing the paper sludge through composting concentrates minerals, but can also have negative environmental impacts because of the increase in the concentration of heavy metals. Despite this concentration, heavy metal content in stabilized compost was well below USEPA's regulated maximum concentration limits (Das et al., 2002). Applying raw residual paper pulp to silty loam increased the concentration of Pb by up to 1.5 mg kg<sup>-1</sup> when 150 Mg ha<sup>-1</sup> were applied (Price and Voroney, 2007). Increases in Cu concentration followed increases in the application rates of the paper pulp residues for a coarse sandy loam. Deinking paper fiber is high in organic matter and Cu (Tandy et al., 2008). Although Cu binds strongly to the deinking paper fiber solid matrix, Cu becomes increasingly bioavailable following paper pulp breakdown after land application. Samples from all Dystric Gleysols indicate an increase in total Cu concentrations resulting from successive deinking paper fiber applications. Even though soil organic matter is regulating Cu bioavailability in soil, no statistically significant relationship was found between soil bioavailable Cu and soil organic matter content. Furthermore, soil bioavailable Cu was not significantly related to plant available Cu and total soil Cu. Spreading deinked paper sludge to farmlands at a rate of 80 Mg ha<sup>-1</sup>year<sup>-1</sup> will exceed United Kingdom soil limits for Cu concentration (50-200 mg Cu kg<sup>-1</sup> depending on pH) after 18 successive applications. Even though short term soil pollution with Cu is not expected, applying deinking paper pulp successively to the same farmland results in Cu contamination.

Ameliorating sandy loam with de-inked and secondary paper sludge mixture deteriorated the physical properties of that soil (Nemati et al., 2000b). Specifically, amending sandy loam under barley and potato cultivation with de-inked and secondary sludge mixture (85 to 15%) significantly decreased wet aggregate stability. It was assumed that increased aeration of the sandy loam compared to the two fine textured soils (silty clay and loamy) resulted in increased microbial activity. Increased microbial activity increased paper sludge degradation and destruction of structure linkages between organic matter, Fe or Al, and mineral particles. As a result, aggregate stability was adversely affected.

Applying paper pulp sludge at rates 90, 110, and 130 t ha<sup>-1</sup> increased the exchangeable sodium percentage (ESP) to 15.52, 14.33, and 14.11%, respectively (Vasconcelos and Cabral, 1993). The significant increase in ESP from increasing rates of pulp mill sludge may result in serious fertility problems, especially if successive applications are made. Using sludge high in Ca and low Na increased ESP because Na showed a higher proportional increase with increasing residual paper pulp application.

Composting raw paper sludge prior land application is practiced occasionally (Tucker and Douglas, 2006), but the end product is not considered more beneficial than the raw pulp and paper sludge (EC, 2001b). Specifically, applying composted de-inked paper sludge on three different soil textural classes (a silty clay, sandy loam, and loamy) did not induce significant changes on wet aggregate stability (Nemati et al., 2000a; Nemati et al., 2000b). In contrast, ameliorating soils (silty clay and loamy) with de-inked and secondary sludge mixture (85 and 15%, respectively) increased wet aggregate stability. Furthermore, composting increases cost, and therefore land application with raw paper sludge is preferable (Vasconcelos and Cabral, 1993).

Direct land application of paper sludges can cause soil N immobilization and induce crop N deficiencies (EC, 2001b). The high C/N ratio of most pulp and paper sludges, combined with high resistance of lignin, cellulose, and hemicelluloses to microbial decomposition, can lead to severe crop losses. Adding extra N fertilizer during the first year of residual paper pulp application can alleviate these yield losses. Vasconcelos and Cabral (1993) reported a decrease in N and P uptake and subsequent decrease in yellow lupine growth when pulp mill sludge applications exceeded 50 t ha<sup>-1</sup>. During the first year of the study, the high C/N and C/P ratios of the applied sludge depressed yellow lupine growth. In the second year, yellow lupine yield was not depressed suggesting that material in the soil had degraded.

## Environmental regulations and effluent standards for the pulp and paper industry Canada

In September 1987, the United States's Environmental Protection Agency discovered dioxins in paper pulp mill effluents, in fish caught downstream from paper mills, and in several paper products (i.e., coffee filters, disposable diapers etc.) (Harrison, 2002). Announcement of the discovery prompted a global discussion about reexamining the operational standards for the pulp and paper industry. Discussion of the issue in three different countries (Canada, The United States, and Sweden) resulted in different approaches and regulatory measures.

In Canada, public awareness of environmental issues related to the pulp and paper industry was further intensified because a 1987 Canadian federal government study revealed that the majority of Canadian paper and pulp mills failed to comply with Canadian federal regulations (Harrison, 2002). In the same year, the Canadian government passed the Canadian Environmental Protection Act (CEPA). Among other substances, dioxins and effluents from paper mills using bleaching were included in CEPA's Priority Substances List for evaluation and

if needed, for regulation. By 1990, CEPA concluded that polychlorinated dioxins and furans were toxic, and hence proposed regulations requiring that the pulp and paper industry reduce dioxin and furan concentrations to non-detectable levels.

Despite their initial intent to propose an Adsorbable Organic Halogens (AOX) limit of 1.5 kg per air-dried metric ton of pulp (kg/ADt), the Canadian Environmental Department (Environment Canada) finally decided not to regulate AOX at all (Harrison, 2002). This complied with Canadian federal scientist's findings showing no relationship between environmental impacts and AOX in mill effluents. Nevertheless, dioxin regulations in paper and pulp mills effluents were finalized in 1992 and were considered equivalent to an AOX standard of 2.5 kg/ADt.

#### Sweden

Pulp and paper mill effluents in Sweden were regulated first under the Environmental Protection Act and then the Swedish Environmental Code (Harrison, 2002). Efforts to regulate chlorinated discharges from pulp and paper mills started well before the United States EPA's dioxin announcement. In the early 1980's the Swedish EPA launched a major research project (Environment/Cellulose I) to determine the impacts of PCB's and other persistent chlorinated organics released from paper mills on the marine environment. In 1985, findings from the research program revealed significant environmental impacts. As a result, the Swedish government began to regulate mills' permits for chlorinated organic discharges. Beginning in 1987, the Swedish EPA issued the Action Plan for Marine Pollution which stated a goal to minimize AOX discharges to 2 kg/ADt by 1992 for all mills using elemental chlorine for pulp bleaching. Following technological developments, AOX targets were tightened further to 1 kg/ADt by 1995 and 0.5 kg/ADt by 2000 for mills using softwood as a raw material. For mills

using hardwood, the targets for AOX were set to 0.5 kg/ADt by 1995 and 0.3 kg/ADt by 2000. However, in response to German consumer demand for chlorine-free paper product the Swedish paper and pulp industry had outperformed the stated regulatory limits on AOX (Harrison, 2002).

In contrast to the North American approach, under the Swedish EPA permit conditions for individual mills were set on a case by case basis (Harrison, 2002). Furthermore, from the 1970's on, the Swedish regulatory approach to prevent environmental pollution focused on internal processes changes rather than the end-of-pipe treatment approach common in North America.

As a result, developing and installing oxygen delignification (bleaching) processes in Kraft mills started in Sweden during the 1970's and became common place in all Sweden mills by the late 1980's (Harrison, 2002). Oxygen bleaching reduces generation of chlorinated compounds. Introducing oxygen delignification technologies was an effort to reduce conventional discharge pollutants from the paper industry. At the same time, few Swedish paper and pulp mills utilized secondary waste water treatment, a standard technology for most American mills.

By 1994 all Swedish Kraft mills had installed oxygen delignification, and had either substituted ClO<sub>2</sub> for chlorine gas (elementally chlorine free or ECF bleaching) or had completely ceased using chlorine for bleaching (totally chlorine free or TCF bleaching) (Harrison, 2002). As a result, AOX levels in paper mill waste water discharges fell from 3.5 kg/ADt in 1988 to 0.4 kg/ADt in 1993. In 1995 industry officials claimed that the average AOX discharge had fallen to 0.1 kg/ADt. This is well below the governmental targets for 2000. In contrast to Canada, the Swedish government continues to regulate AOX discharges from pulp and paper mills.

Furthermore, Swedish EPA never set specific limits on dioxins since the industry resolved the issue on its own.

#### The United States

In the United States, the Water Clean Act regulates paper and pulp mill discharges (Harrison, 2002). This is achieved at two levels. On the one hand, EPA issues technology-based guidelines for comparable sources and the guidelines are then incorporated into individual facility permits. Standard settings for toxic pollutants are based on best available technology that is economically achievable. On the other hand, state and tribal governments can establish more stringent standards to meet local water quality objectives.

In 1988, EPA agreed to consider regulating dioxins from paper mill effluents and other sources (Harrison, 2002). In 1992, EPA released the "cluster rule" where regulations for air, water, and sludge for the paper and pulp mill industry are merged together. In the 1993 Federal Register, the agency proposed an AOX limit for paper and pulp Kraft mill effluents of 0.156 kg/ADt. Achieving the proposed limit would require Kraft mills to move towards ECF bleaching plus oxygen delignification. Considering the proposed limit not economically achievable, the American paper and pulp industry opposed the proposed AOX limit. The American Forest and Paper Association supported complete substitution of ClO<sub>2</sub> for elemental chlorine but no oxygen delignification. In November of 1997, EPA announced its decision to favor the latest option, stating that oxygen delignification technology was not economically achievable. Surprisingly, the decision came at a time when one third of American paper and pulp Kraft mills had already installed oxygen delignification technology. Furthermore, at the same time in Sweden all Kraft mills were utilizing oxygen delignification and the vast majority of mills was using even more advanced technology.

Nowadays, standards for paper and pulp mill discharges are set for 2, 3, 7, 8-tetrachlorodibenzo-p-dioxin, 2, 3, 7, 8-tetrachlorodibenzofuran, chloroform, 12 chlorinated phenolics, and AOX (Harrison, 2002). The AOX average monthly limit for existing Kraft mills is 0.623 kg/ADt, while new mills must install oxygen delignification technology and the AOX limit for them is set to 0.272 kg/ADt.

#### **CHAPTER 3**

### MATERIALS AND METHODS

# **Study locations**

This study reports partial data and results from a 4-year research project (2007-2011) directed by Dr. Athanasios Gertsis and funded by MEL Macedonian Paper Mills, S.A., Greece. The study took place near Thessaloniki, Greece (Figures 3.1 and 3.2) at two fields with different soil textural classes: 1) a clay loam located at American Farm School at Thermi, (Figure 3.3), (N40° 33′ 58.67″, E22° 59′ 27.58″) and 2) a sandy loam located at Kolchiko, (N 40° 43′ 27.48″, E23° 08′ 18.71″) near Lagada, (Figure 3.4). Each field was 0.1 ha in area and was divided into 16 experimental plots. The experimental plots were each 3 m in width and 10 m in length (total area of 30 m²). To reduce side effects from residual paper pulp applications on adjacent experimental plots, each plot had 2 m-wide borders within the replications, and 1.5 m-wide borders between replications.

The experiment consisted of four treatments. Each treatment was replicated four times for a total of 16 plots. Each treatment was randomly assigned to one of the four plots within each replication (Figure 3.5).

### Recycled residual paper pulp

The recycled residual paper pulp (hereafter referred to as RRPP) used in the study came from the precipitation tank of Macedonian Paper Mill S. A. at Gefyra, Greece, the largest cardboard-box producing company in Greece. The RRPP was air-dried for a period of 15 days. The dried pulp was then finely ground at the CaO HELLAS S.A. facilities in Thessaloniki,

Greece (a company producing agricultural lime and other calcium carbonate products used in for constructions) resulting in pulp particles ranging in size from 2-4 mm. The ground pulp was then placed in 45L plastic bags for transport, storage and eventual application to the field. Samples of the pulp were analyzed by independent laboratories in the USA, Germany, and Greece and results are provided in Tables B.1 to B.3.

The pulp was hand-applied to the soil surface of the designated experimental plots at the beginning of the first and second growing seasons using the following four application rates: 0, 1, 2, and 4% volume/volume (v/v) calculated for the top 0.15-m soil depth (or 0, 1.5, 3, and 6 L m<sup>-2</sup> or 0, 15, 30, and 60 m<sup>3</sup> ha<sup>-1</sup>), The application rates can be converted to dry Mg ha<sup>-1</sup> using an average material bulk density (BD) of 0.637 Mg m<sup>-3</sup>, which corresponds to 0, 9.6, 19.2, and 38.4 dry mass Mg ha<sup>-1</sup>, respectively. The rates are expressed in a dry volume/volume basis for consistency in both soil types (equal amounts of RRPP was applied for each treatment per unit of soil volume). A rotary tiller was used to incorporate the pulp into the topsoil (0.15-m soil depth). Soil samples were collected twice to this depth each year – at the beginning and end of each growing season. Samples were air dried, sieved through a 2-mm sieve and stored in the laboratory room at room temperature (20-25°C).

# Planting schedules and crop species cultivated

A sequence of three crops were grown at each experimental site during each growing season. Planting dates for all three crops and locations are presented in Table 3.1. In the first year (2008), cabbage (*Brassica oleracea* L., var. Potomak and Grandslam) and lettuce (*Lactuca sativa* L. var. Justine-butterhead type) seedlings were planted using a 1-m row spacing and a 0.5-m plant spacing within the row, and corn was planted using a 0.75-m row spacing and a 0.1-m plant spacing within the row. The cabbage was followed by lettuce, and at the end of the first growing

season sweet corn (*Zea mays* L. var. *saccharata* Legend F1 hybrid) was sown with a 4-row pneumatic planter. In the second growing season, market shortages in cabbage seedlings delayed crop planting and shifted lettuce crop cultivation to the beginning of the growing cycle. *Lactuca sativa* L.var. Parris Island-COS type was grown for the 2009 season. The same sweet corn variety was planted during the second experimental year. However, the cabbage was removed from the field before reaching maturity because of high seasonal temperatures, and therefore cabbage yield data are available only for the first growing season.

During both years (2008), LISA (Low Input Sustainable Agriculture) cultivation practices were used. During the first year, an organic pesticide based on *Bacillus thuringiensis* (Bactospeine ®, a.i. Bt) was used for the control of cabbage caterpillars on both experimental sites. No fertilizers and synthetic pesticides were applied. Although LISA practices were followed when possible during the second year, it was necessary to use small amounts of fertilizers and some synthetic herbicides (Treflan ®, a.i. trifluralin) (in one case only at Kolhiko, 2008). At planting, 5 kg/ha of 11-15-15 start-up fertilizer was applied on both fields at the beginning of the second growing season.

In both years, the cabbage and lettuce crops were irrigated by drip irrigation lines with emitters spaced at 0.5-m intervals and with an output of 4 L h<sup>-1</sup>. The sweet corn was irrigated by drip irrigation and by some supplementary overhead irrigation with a watering gun, only during the first (2008) season. The following year (2009), all crops at AFS site were irrigated with drip irrigation, while at Kolhiko a supplementary overhead irrigation was again provided for the sweet corn crop.

Cultivation practices included manual and mechanical weeding. A small rotary tiller was utilized for the mechanical weeding in the cabbage and lettuce crops, and a three-row cultivator for weeding in the sweet corn crop.

# Soil chemical and physical properties

Soil properties measured were: 1) soil reaction (pH), 2) saturated electrical conductivity, 3) bulk density and porosity, 4) volumetric water content, 5) apparent electrical conductivity, 6) temperature, 7) organic carbon, 8) heavy metals, 9) total N, 10) and NO<sub>3</sub>-N and NH<sub>4</sub>-N. Top soil (0-0.15 m) physical and chemical properties were measured in the Agronomy & Soils Laboratory of Perrotis College, Thessaloniki, Greece. Additional analyses (CaCO<sub>3</sub>, P<sub>2</sub>O<sub>5</sub>, K<sub>2</sub>O, total N, NO<sub>3</sub>-N, NH<sub>4</sub>-N and heavy metals) were also provided from the Department of Agrochemistry and Plant Physiology, Faculty of Agriculture, University of Belgrade, Serbia (Professor Valdo Licina).

Soil reaction (pH) and saturated electrical conductivity (ECs) were measured in the laboratory using an 1:1 mixture of soil to distilled water (v/v) with a combination pH-EC electrode (Model HANNA HI 9811) calibrated each time of measurements with standard solutions of pH and EC. Soil bulk density (BD) and porosity were measured taking undisturbed soil samples with the "cylinder method". Undisturbed top soil cores were collected with 100 cm<sup>3</sup> stainless steel cylinders. Then, the soil cores were taken to the laboratory and oven-dried for 24-48 hrs to determine net oven-dry mass of soil. Bulk density was calculated as the ratio of oven dry mass/soil volume. Porosity was calculated based on BD from the relationship: Porosity=1-Bulk density/Particle density. Particle density was taken equal to 2.65 Mg m<sup>-3</sup>. The cylinders were pressed into the soil to a depth of 0.15-m. Soil organic carbon was measured with the Walkey-Black wet combustion method (Walkey and Black, 1934; Walkey, 1947). Periodical

measurements in the top soil (0 to 0.15-m) for soil volumetric water content (VWC), apparent electrical conductivity (ECa), and temperature were performed with the WET <sup>®</sup> sensor and the HH2<sup>®</sup> datalogger (Figure 3.6).

# **Agronomic measurements**

Plant properties and agronomic characteristics measured were: 1) leaf chlorophyll level (lettuce and corn), 2) yield (fresh weight of lettuce, cabbage, and corn ears). Relative leaf chlorophyll level (expressed as SPAD units) was measured in five representative plants from the middle experimental row with a Minolta SPAD 502 ® leaf chlorophyll meter (Figure 3.7). Each of the five readings was the average of three leaf readings per plant (lettuce) taken at various leaf ages (a low, a middle, and an upper leaf in the canopy were sampled and averaged at each plant). For sweet corn, SPAD measurements were taken at the same day of the two harvestings in 2009, with three average measurements from each of the ten plants where the ear was harvested, within each experimental plot. In lettuce, SPAD units were measured in the last harvest set.

Fresh yield was recorded as the average of five harvested plants for the cabbage and lettuce crops and ten randomly selected ears of sweet corn crop from the middle planted line of each experimental plot. All samples were harvested from the middle experimental row to avoid any edge effects.

#### Other data and observations recorded

Automated weather stations were installed adjacent to each of the study sites and recorded data at hourly intervals. Weather data at 2 m height collected at each site were: 1) air temperature and relative humidity (RH%), 2) rainfall (mm), and 3) wind speed and direction,. Summary weather data recorded for each crop growing season are reported in Appendix C (Tables C.1 and C.2; and Figures C.1 to C.4).

# **Statistical analyses**

The field experiment was analyzed as a Randomized Complete Block Design (RCBD), because the four treatments were assigned randomly to each of the four replications or blocks. "Block" is the preferred statistical term since it avoids confusion with the term "replication" which is used when the analysis follows the completely randomized design (CRD). The main objective of the RCBD is to "keep the variability among experimental units within a block as small as possible and to maximize differences among blocks. If there are no block differences, this design will not contribute to precision in detecting treatment differences" (Little and Hills, 1978). The units in a block are as uniform as possible so that the observed differences should be primarily due to treatment effects (Steel and Torrie, 1980). The borders left around each experimental plot further facilitated the RCBD analysis. The 4 rates of RRPP were considered as continuous-nominal variables as well as the blocks, for the statistical analyses.

JMP® 7 (JMP®, 2007) software was used for the statistical analysis of collected data. Statistical analyses of treatment effects on soil and agronomic properties measured was carried out with one-way ANOVA by blocking treatments. All pairs mean comparison was done with Student's t-test. Furthermore, simple linear regression analysis with treatments and blocks in the constructed model was also used for data analysis.

Table 3.1 Planting dates for *Zea mays* L. *saccharata* (sweet corn), *Brassica oleracea* L. (cabbage), and *Lactuca sativa* L (lettuce) for 2008 and 2009.

Crop species and varieties	Planting date	e
	AFS	Kolhiko
Zea mays L. saccharata var. Legend F1 hybrid	2 July 2008	25 June 2008
Zea mays L. saccharata var. Legend F1 hybrid	20 May 2009	9 May 2009
Brassica oleracea L. var. Potomak and. Grandslam	6 September 2007	18 September 2007
<i>Brassica oleracea</i> L. var. Potomak and. Grandslam	Discontinued	Discontinued
Lactuca sativa L., var. Justine	13 March 2008	12 April 2008
Lactuca sativa L., var. Parris Island	13 November 2009	15 November 2009

Notes: Zea mays L. saccharata var. Legend F1 hybrid was planted on both fields in 2008, and 2009.

*Brassica oleracea* L. var. Potomak and var. Grandslam were planted in 2007/2008 growing season; *Brassica oleracea* L for 2008/2009 growing season was discontinued. *Lactuca sativa* L, var Justine-butterhead type planted in 2008; *Lactuca sativa* L., var. Parris Island-COS type planted in 2009)

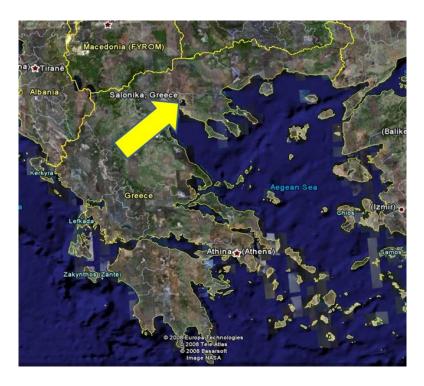


Figure 3.1 Map of Greece. Arrow indicates area of Thessaloniki (from Google earth).

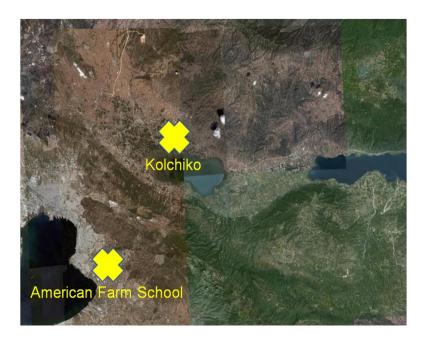


Figure 3.2 AFS and Kolhiko field locations (from Google earth).



Figure 3.3 American Farm School field after RRPP application, 10-21-2008.



Figure 3.4 Soil cultivation during 1<sup>st</sup> growing season at Kolhiko field.

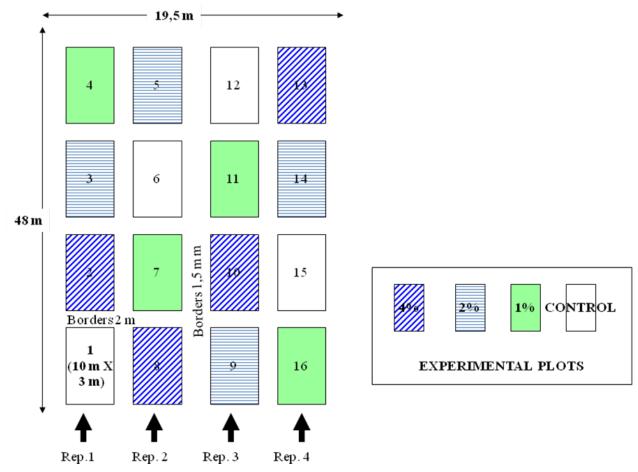


Figure 3.5. Experimental design for AFS and Kolhiko sites (2007-20011).



Figure 3.6 The WET sensor and the HH2 datalogger.



Figure 3.7. The leaf chlorophyll meter (SPAD Minolta 502).



Figure 3.8 Automated Weather Stations were installed in each soil.

#### **CHAPTER 4**

### **RESULTS AND DISCUSSION**

### SOC analysis for AFS and Kolhiko sites

Ameliorating soil with RRPP at Kolhiko site increased the mean %SOC at the end of the first growing season (2008) for all treatments (1, 2, and 4% v/v RRPP application rates) compared to the control where no RRPP was applied (Figure 4.1).

The 2 and 4% RRPP application rates resulted in statistically significant increases in %SOC at a 0.05 level of significance (Table 4.1). By the end of the first year, the 1% treatment in the sandy loam had increased %SOC, as well, though the increase was only numerically higher.

Simple linear regression analysis for %SOC at Kolhiko (2008) with treatments as factors (nominal) and replications as blocks (nominal) resulted in a coefficient of determination of 0.488 (Table 4.2). Therefore, only 48.8% of the total variation in the SOC is explained by the RRPP application rates. The predictive value of the model is not significant at α=0.05, as shown from the overall p-value=0.1324 (Table 4.3), although the intercept and slope (treatment coefficients) are significantly different from zero (p-values<0.05; Table 4.4).

Data analyses (One-way ANOVA by blocking replications) for soil (sandy loam) samples taken in fall 2009 revealed a mean increase in SOC for all application rates (Figure 4.2).

Applying RRPP at the 4% rate for a second consecutive year at the Kolhiko site resulted in a significant increase in SOC with respect to control (Table 4.1). This finding agrees with reported

increases in soil total C from residual paper pulp applications on coarse loam and loamy sand (Foley and Cooperband, 2002; Price and Voroney, 2007).

In results for fall 2009, simple linear regression analysis for SOC at Kolhiko showed  $R^{2=}0.506$  (Table 4.5). Therefore, 50.6% of the total observed variability in SOC is explained by the linear relationship between SOC and pulp application rates. For data collected in 2009, the coefficient of determination ( $R^2=0.506$ ) for SOC is a slightly higher compared to SOC results for the first ( $R^2=0.448$ ). The analysis of variance reveals p-value=0.078 (Table 4.6), and therefore, the overall simple linear model will not predict significant changes in SOC at different RRPP application rates.

On the first growing season at the AFS field (clay loam), application of RRPP did not result in significant increases in SOC at any of the application rates (Figure 4.3; Table 4.1). A non significant increase in SOC at the AFS field may be explained by the larger initial content of SOC at this site compared to the Kolhiko site (1.246 SOC and 0.782 SOC respectively).

Simple regression analysis for the data collected on the 1<sup>st</sup> growing season from the clay loam soil (SOC vs. Treatments with replications as blocks) resulted in R<sup>2</sup>=0.204 (Table 4.7). Therefore, only 20.4% of the variation in SOC observed in the field is explained by the linear relationship between SOC and RRPP application rates. Furthermore, the analysis of variance (Table 4.8) shows that the specific model does not adequately fit the data (p-value>0.6). As a result, for the clay loam, changes in SOC are not predicted for different pulp treatments by simple linear relationship.

On the second experimental year, one-way ANOVA for SOC data in the clay loam indicates statistically significant increases for 2 and 4% v/v RRPP application rates with p-value<0.05 (Table 4.1). The data show 0.315 and 0.266% increases in SOC content for 4 and 2%

v/v pulp application rates, respectively, by the end of 2009. These results show the potential value that RRPP has as soil amendment and agrees with results of Beyer et al. (1997), which showed that SOC was increased by application of paper mill pulp (30 and 60 t/ha combined with 70 and 140 m³ of cattle slurry) in a sandy soil in Germany; however this effect disappeared within one year. Overall in their 3-year project, Beyer et al. (1997) concluded that one application of paper pulp within a 3-5 year crop rotation will not affect the soil ecosystem in a negative way.

Simple regression analysis (blocking replications) for SOC data at the AFS field (2009) indicates strong linear relationship for SOC and pulp application rates (Table 4.9). Specifically, 84.5% of the observed variability in SOC is explained by RRPP application rates. Furthermore, the analysis of variance (Table 4.10.) indicates that the overall model predictability is significant at a level of significance  $\alpha$ =0.05 (model p-value=0.0002). All model parameters, with the exception of blocked replication 1, are significant (Table 4.11). Therefore, applying pulp for two years to a clay loam such as the one used in this study would be expected to result in a 0.08% increase in SOC content for every unit increase (% v/v) in RRPP application rate.

Figure 4.5 indicates that amending soil for two consecutive years at the sandy loam experimental site resulted in SOC increase for all treatments. Furthermore, a cumulative trend in SOC is observed. At the end of the 1<sup>st</sup> growing season, SOC at Kolhiko site increased by 0.176, 0.383, and 0.324% for 1, 2, and 4% v/v pulp application rates (Table 4.1). In fall 2009, SOC in the sandy loam had increased further. More specifically, SOC analysis indicates 0.326, 0.388, and 0.539% increase at 1, 2 and 4% v/v pulp application rates if compared to control from the first year. In 2008, increases in SOC were not directly proportional to RRPP application rates, but still the two highest application rates (2 and 4% RRPP v/v) were statistically significant at

 $\alpha$ =0.05. Furthermore, in 2009 the increase in SOC for the sandy loam was directly proportional to RRPP application rates, though only the 4%v/v paper pulp application rate was significant (p-value<0.05).

Conditioning the clay loam soil with RRPP in 2008 (1<sup>st</sup> growing season) did not result in significant increases (Table 4.1; Figure 4.6). After two years of pulp application, SOC content at the AFS site increased by 0.194, 0.363, and 0.412% compared to 1<sup>st</sup> year unamended plots. Furthermore, treatments 2 and 4% v/v were statistically significant with p-value<0.05. Therefore, two successive application rates were necessary to observe significant changes in SOC in the clay loam soil, while in the sandy loam significant differences in SOC were observed with the first year's applications. Textural effects on N immobilization were suspected as reported in other studies (Aitken et al. 1998).

# pH measurements at the AFS soils laboratory

One-way ANOVA by blocking replications indicates that soil pH means were not statistically significant (Table 4.12) between the different treatments at the AFS site for 2008. Therefore, one year application of RRPP did not affect soil pH at the clay loam.

The summary of Fit, from the simple linear regression (with replications as blocks), showed  $R^2$ =0.492 (Table 4.13). This suggests that less than 50% of the variability in pH is explained by RRPP application rates (2008) for the clay loam. Furthermore, the overall linear regression model (Table 4.14) does not fit the data well (p-value=0.09) at a level of significance  $\alpha$ =0.05. Therefore, there is no linear relationship between soil pH and RRPP application rates. This conclusion confirms our findings from one-way ANOVA analysis of pH vs. treatments (by blocking replications) discussed earlier.

Soil pH data analysis with one-way ANOVA (by blocking replications) revealed insignificant changes in soil pH at the clay loam site after two consecutive pulp applications at rates of 1, 2, and 4% v/v (Table 4.12). As a result, ameliorating clay loam with two successive doses at 1, 2 and 4% v/v rates should not shift soil pH dramatically. This makes RRPP a suitable soil amendment material for clay loams that require improvements in soil chemical and physical properties other than pH (i.e., increase in SOC content).

One—way ANOVA conclusions were confirmed from the simple linear regression analysis with blocks as well. More specifically, an R<sup>2</sup> =0.097 from Table 4.15 indicates that conditioning clay loam with RRPP for 2<sup>nd</sup> consecutive year accounts for less than 10% of the pH variability observed at the field. The large p-value (Table 4.16) indicates that the simple linear regression model does not fit the data well, and therefore pH is not linearly related to pulp application rates at the AFS site (end of second season).

Applying RRPP at the beginning of the first growing season increased the mean pH at the sandy loam site for all application rates (1, 2, and 4% v/v) as shown in Figure 4.7. Treated plots have means above the grand mean which is indicated with the horizontal gray line. Furthermore, comparison of means with Student's t-test indicates a statistically significant increase in pH for all treated plots (1, 2, and 4% v/v) compared to control (Table 4.12) at a level of significance α=0.05. The largest raise in soil pH at the Kolhiko experimental site was observed for the 2% v/v rate, followed by the 1 and 4% v/v application rates, respectively. But these differences were only arithmetical and cover a pH range from 6.9 to 7.0. Generally, the pH buffering capacity of the sandy loam is lower compared to clay loam textural class soil. Therefore, sandy loams are more susceptible to pH changes when materials with acidic or liming properties are added.

Statistically significant soil pH changes obtained at the sandy loam site were expected. Thus,

close monitoring of soil pH is required when yearly doses of the RRPP are applied to sandy loams.

Simple regression analyses (with replications treated as blocks) of data (Kolhiko, 2008) gave moderate to low  $R^2$ =0.523 (Table 4.17) Therefore, only 52.3% of the total pH variability observed in experimental plots is accounted by pulp application rates. The analyses of variance for pH (Table 4.18) showed a large p-value=0.066. As a result, the predictability of the overall linear model is insignificant at level of significance  $\alpha$ =0.05.

Applying pulp in sandy loam for a second consecutive year resulted in a mean pH increase in all treated plots (Figure 4.8). Means of all rates (1, 2 and 4% v/v) were above the grand mean. Data analysis with one-way ANOVA and replications treated as blocks indicates a significant increase in soil (sandy loam) pH for all the treatments where RRPP was applied (Table 4.12).

At the end of the second year, the sandy loam pH had changed from slightly acidic to slightly alkaline. RRPP from Macedonia Paper Mill SA contains CaCO<sub>3</sub> (Table B.2) which reacts with CO<sub>2</sub> and H<sub>2</sub>O in the slightly acidic soil to produce Ca<sup>+2</sup> and HCO<sub>3</sub><sup>-</sup> ions (Brady and Weil, 2008). Calcium bicarbonates have liming capacity, since they are reactive with residual and exchangeable soil acidity. More specifically, Ca<sup>+2</sup> replaces H<sup>+</sup> and Al<sup>+3</sup> ions on the colloidal complexes, followed by insoluble Al(OH)<sub>3</sub> formation, and CO<sub>2</sub> emission to the atmosphere. Consequently soil solution pH rises. Therefore, long term application (more than 2 consecutive years) to a slightly acidic sandy loam would require close pH monitoring to keep soil reaction at optimum level for crop cultivation.

For the second experimental year, a simple regression analysis with replications as blocks reveals  $R^2$ =0.500 at Kolhiko field (Table 4.19). There is no strong linear relationship between the

observed variability in sandy loam pH for the  $2^{nd}$  year and pulp application rates. The predictability of the overall model is insignificant since the p-value is larger than level of significance  $\alpha$ =0.05 (Table 4.20).

# EC measurements on AFS and Kolhiko experimental sites

Ameliorating clay loam with pulp resulted in insignificant changes in soil ECs (Table 4.21) for the first year. The largest mean ECs was measured at the 1% v/v plots, but this value was well below the salinity limiting threshold of 4 dS m<sup>-1</sup>.

Simple linear regression explained 45.4% of the variation observed in clay loam ECs (Table 4.22). As a result, conditioning soil with RRPP did not account for variability in soil ECs. The predicting value for the overall model is insignificant as indicated by the large p-value=0.125 (Table 4.23).

Similar trends for soil ECs were found at the end of the second growing seasons (fall 2009) at the AFS experimental area. One way ANOVA by blocking replications resulted in insignificant changes in mean ECs (Table 4.21).

Comparing ECs means from 2008 and 2009 (Table 4.21) we observed an increase in ECs throughout the entire experimental plot (AFS site). Interestingly the largest arithmetical mean value in soil ECs was measured for the control treatment (1.599 dS m<sup>-1</sup>). Nevertheless, all treatments showed an increase in mean ECs, but they were still well below the limiting salinity threshold of 4 dS m<sup>-1</sup>. This raise in the lab measured ECs is not explained from the RRPP application rates on the second experimental season alone. Other factors, such as high field variability, fertilization (fertilizer was applied only during the second experimental year), and most importantly change in irrigation pattern during the second experimental year may be responsible for increased ECs values. More specifically, at the AFS site the field for the entire

second growing season was placed under drip irrigation, whereas on the first season the sweat corn was irrigated both by drip irrigation and by cannon irrigation systems. Less irrigation water input for the second growing season probably provided inadequate drainage and therefore salt accumulate in the top 0.15 m.

Simple linear regression explained only 34.5% of the soil ECs variation observed at the AFS site (Table 4.24), and the overall model predictability for salts concentration is insignificant (p-value>0.05) (Table 4.25).

ECs results for the sandy loam (end of 1<sup>st</sup> growing season), indicate that there were insignificant changes in ECs means related to the RRPP application rates (Table 4.21). Interestingly, 4%v/v application rate gave the lower ECs value (0.26 dS m<sup>-1</sup>), though, the value was close to the results of the other application rates. Analysis of the RRPP material showed consistently low ECs (0.57 to 0.934 dS m<sup>-1</sup>), and therefore, one year application may not change significantly the soil's ECs (Table B.2).

Simple regression analysis of ECs data (Kolhiko, 2008) with blocking (replications) effect did not explain ECs variation throughout the field, since R<sup>2</sup>=0.13 (Table 4.26). Predictability of the model is insignificant as indicated by the large p-value (Table 4.27).

ECs means in fall 2009 (Figure 4.9) at the Kolhiko site are above the grand mean for 1 and 2% v/v RRPP rates, whereas treatments 0 and 4% v.v. showed means below the grand mean. Conditioning sandy loam for a second consecutive year resulted in statistically significant increases in mean ECs for application rates 1 and 2 % v.v. of RRPP (Table 4.21). The larger application rate (4% v/v) showed ECs values that were not significantly different from the control and 2% v/v rates. Nevertheless, two years of consecutive pulp application on a sandy loam raised

the ECs, but to levels well below the 4 dS m<sup>-1</sup> which is an established threshold for salinity considerations.

Simple linear regression analysis for ECs data (Kolhiko,  $2^{nd}$  growing season) showed small  $R^2$ =0.345 (Table 4.28). Furthermore, analysis of variance (Table 4.29) for the simple linear regression model reveal insignificant model predictability (p-value=0.875) at level of significance  $\alpha$ =0.05.

Applying RRPP pulp on two different soils (clay loam, and sandy loam) showed that two years of consecutive RRPP application rates (1, 2, and 4% v/v) did not significantly affect the ECs at the AFS site (clay loam). Statistically insignificant alteration on sandy loam ECs was observed at the end of the first growing season. In fall 2009, soils sample analyses from the Kolhiko site indicated statistically significant increases in the ECs for 1 and 2 application rates (0.888 and 0.813 respectively). In both sites, pulp application at rates of 1, 2, and 4% v/v did not raise soil's ECs above the limiting salinity threshold (4 dS m<sup>-1</sup>).

# Total N measurements on AFS and Kolhiko sites

Analysis of data (One-way ANOVA) gathered from the AFS experimental area indicates that soil conditioning with RRPP did not significantly increased Total N content (Table 4.30) at level of significance  $\alpha$ =0.05. Pulp analysis at different laboratories for C/N varied significantly from 32.8 up to 60.1 (Table B.2). Nevertheless, pulp decomposition was expected to result in N immobilization right after application because RRPP had high C/N ratio (C/N>24/1). At the end of the 1st growing season, it seems that N was not immobilized nor mineralized, at statistically significant rates.

Simple linear regression (blocking replications) did not explain satisfactorily the variation in Total N% after one year of pulp application (Table 4.31). Large p-value=0.831 indicates that

the model (simple linear regression) predictability in Total N% in the soil is not significantly related to pulp application. Therefore, pulp N nutritive value is either low, and/or any N mineralized during pulp decomposition was either leached to lower soil horizons and/or assimilated into phytomass. A second consecutive application of pulp to the clay loam did not change significantly total soil N (Table 4.30).

For 2009, simple linear regression for Total N at the AFS site (blocking replications) had low  $R^2$  (0.300), indicating that Total N was weakly related to pulp application rates (Table 4.33). The overall model has p-value> $\alpha$ =0.05 (Table 4.34), and therefore, it is not useful in predicting Total N concentration related to pulp application rates at the clay loam site.

Data analysis (One-way ANOVA, blocking replications) revealed that RPPP application for the first year did not significantly alter Total N concentration in the sandy loam for any treatment (Table 4.30).

R<sup>2</sup> for Total N% at Kolhiko for 2008 was low (0.125), and therefore there is very low linear relationship between Total N and pulp application rates (Table 4.35). As p-value=811 indicates in Table 4.36 the linear model did not fit the data adequately. Its predictability in sandy loam Total N concentration related linearly to RRPP application rates is insignificant.

Conditioning sandy loam with RRPP for two consecutive years did not affect Total N concentration, as indicated from one-way ANOVA analysis (Table 4.30). Ammonium volatilization from a nearby small manure pile (15 m northwest of the plots on the 4<sup>th</sup> replication block), may explain Total N increase throughout the entire experimental area. Fertilization definitely contributed to raise N concentration in all experimental plots.

Furthermore, simple liner regression (Table 4.37) showed small coefficient of determination ( $R^2$ =0.300). Consequently, only 30% of the total variability in N concentration is

explained from different pulp application rates. From Table 4.38 we conclude that the predictive value of the model (simple linear) for Total N concentration is insignificant (p-value=0.373).

Total N analysis at all sites for both years did not indicate any significant change in soil Total Nitrogen content for all treatments (0, 1, 2, and 4% v/v RRPP). This finding shows that although the material has high C/N (>24/1) no N immobilization occurred. The results agree with the findings of Price and Voroney (2007), who reported that amended soils with high C/N ratio de-inked paper sludge did not affect soil total N after 2 years. A decrease in total soil N was observed only in a silt loam as a result of increasing rates of paper sludge treatments (50, 100, and 150 Mg ha<sup>-1</sup>). Furthermore, increasing rates and successive applications of secondary sludge on a sandy loam raised NO<sub>3</sub>-N concentrations at the top 0.15 m. In contrast, Douglas et al. (2003) studied the effects of five non-agricultural organic wastes (one was paper-mill sludge) on soil composition, grass yield and grass N use in a 3 years field experiment. They concluded that all wastes had no unfavorable effects on soil. The paper mill sludge caused N immobilization and their results demonstrated clear differences in the ability of the applied wastes to provide cropavailable N.

#### C/N ratio at AFS and Kolhiko site

Ameliorating clay loam did not significantly affect C/N at level of significance  $\alpha$ =0.05, as indicated from C/N mean comparisons (Table 4.39). This result agrees with findings in SOC (both analyses at AFS and University of Belgrade, Serbia soil labs showed insignificant SOC changes) and Total N (non significant) for the first year of RRPP application at the AFS site.

Simple linear regression with replications as blocks (Table 4.40) showed  $R^2$ =0.157, and therefore 15.7% of the C/N variability is explained from the model. Large p-value (Table 4.41) indicates poor predictability for the overall model.

In fall 2009, the C/N ratio had not changed significantly at the AFS site, as indicated from one-way ANOVA (blocked replications) (Table 4.39). Only arithmetical increases are observed for application rates 2, 1, and 4% v/v compared to control. Long term research is likely required to determine if the pulp can significantly increase soil carbon, and subsequently alter C/N ratio.

Simple regression analysis (Table 4.42), for C/N data retrieved from clay loam soil in 2009 resulted in  $R^2$ =0.455, which is moderately low, though larger than the  $R^2$ =0.157 obtained in 2008. The p-value (Table 4.43) is far larger than  $\alpha$ =0.05, and therefore, the C/N ratio predictive value of the model (simple linear) is not significant.

At the Kolhiko site, ameliorating soil with pulp did not significantly affect C/N ratio means (Table 4.39). Simple linear regression for 1<sup>st</sup> growing season C/N data revealed a large coefficient of determination (R<sup>2</sup>=0.731), and therefore, 73.1% of the total variability in soil C/N is explained from pulp application rates (Table 4.44). Small p-value=0.004 (Table 4.45) indicates that the overall model is significant. Though, parameter estimates for C/N (Table 4.46) revealed that RRPP application rates are insignificant, while the intercept (initial C/N ratio) and block 1 are significant in predicting C/N in sandy loam. This confirms the conclusions from one-way ANOVA, which indicates non significant differences in mean C/N for 1, 2, and 4% v/v rates.

At the end of the second growing season, one-way ANOVA analysis showed arithmetical increases at sandy loam C/N means, which were directly proportional to RRPP application rates (Table 4.39), but not statistically significant at level of significance  $\alpha$ =0.05. Seasonal increase in mean C/N ratio for all treatments is probably related to crop residues from 1<sup>st</sup> growing season.

Simple linear regression for data on C/N ratio (Kolhiko, 2009) gave small R<sup>2</sup> (Table 4.47). Overall model predictability for C/N levels related to pulp application rates is insignificant

at level of significance  $\alpha$ =0.05 (Table 4.48). We did not find simple linear relationship among C/N in the soil and pulp rates.

# SOC analyses at AFS and Kolhiko site

The SOC analyses reported in this section were conducted at the Department of Agrochemistry and Plant Physiology, Faculty of Agriculture, University of Belgrade, Serbia.

At the end of the 1<sup>st</sup> growing season soil (clay loam) samples were analyzed for SOC. Data analysis with one-way ANOVA (using replications as blocks) indicates no significant differences in SOC for 2008 soil samples (Table 4.49). Therefore, first year pulp application rates did not change SOC at the clay loam.

Simple linear regression revealed very low R<sup>2</sup>, and therefore the variability in SOC is not linearly explained by the RRPP application rates (Table 4.50). In addition, analysis of variance for the overall model shows large p-value=0.937 (Table 4.51). As a result, the predictive model value for SOC vs. pulp treatments is insignificant.

Data analysis for SOC in fall 2009 (One way ANOVA with replications as blocks) showed insignificant increase in SOC in the clay loam for all plots treated with pulp (Table, 4.49). At the end of the second growing season, mean SOC for plots ameliorated with 1, 2, and 4% v/v increased by 0.21, 0.31, and 0.29% respectively compared to 1<sup>st</sup> growing season control. Similar results observed for SOC analysis at AFS soil lab, though SOC increase was directly proportional to pulp application rates.

Simple linear regression model for SOC data in fall 2009 (AFS site) have  $R^2$ =0.643 (Table 4.52). Ameliorating soil with RRPP accounts for 64.3% of the variability in SOC observed in the soil in fall 2009. Model usefulness is significant at level of significance  $\alpha$ =0.05 (Table 4.53). The linear model

AFS SOC 2009=1.439+0.063\*RRPP rate ( $R^2$ =0.643, p-value=0.016)

is significant, and shows that for every 1% increase in RRPP application rate we should expect 0.063% increase in SOC at the clay loam soil (Table 4.54).

For the sandy loam (2008), one-way ANOVA (with replications as blocks) indicates that application of pulp did not affect SOC significantly at any of the rates. Simple linear regression for SOC vs. treatments (Table 4.55) has very low R<sup>2</sup>=0.083. Analysis of variance for SOC for 1<sup>st</sup> growing season at Kolhiko reveals large p-value, and therefore the overall model's predictive value is insignificant (Table 4.56).

At the end of the second growing season, mean comparison (One-way ANOVA with blocking replications) for sandy loam plots treated with RRPP showed insignificant differences at  $\alpha$ =0.05 (Table 4.49). Furthermore, SOC at the sandy loam was 0.525, 0.470, and 0.365% for 1, 4, and 2% v/v RRPP application rates respectively. Long term research is required to determine if the numeric increase can become statistically significant, and thus, evaluate material's usefulness as soil adding carbon amendment.

Simple linear regression analysis for data gathered on the second year at the sandy loam experimental area gave a small  $R^2$ , thus, less than 22% of the SOC variability is accounted for paper pulp application rates (Table 4.57). Analysis of variance resulted in large p-value=0.565, and therefore model predictability in SOC for sandy loam treated with RRPP is insignificant (Table 4.58).

# Bulk density analysis for AFS and Kolhiko site

Ameliorating clay loam with RRPP in 2008 resulted in a statistically significant decrease (p-value<0.05) in soil bulk density (BD) for plots treated with 4% v/v pulp rates (Table 4.59). A mean BD of 1.098 Mg m<sup>-3</sup> was measured for the largest application rate, when 0, 1, and 2% v/v

pulp treatments shown mean BD of 1.198, 1.194, and 1.177 Mg m<sup>-3</sup> respectively. The decrease in the top 0.15 m soil's bulk density at the AFS site is probably caused from an arithmetic increase in SOC for pulp treated plots (AFS SOC analysis, Table 4.1). At the end of the 1<sup>st</sup> growing season, a statistically significant decrease in soil BD for 4% v/v rates indicates: 1) increased porosity, 2) improved root growth environment, 3) improved soil aeration, and 4) increased water infiltration (Brady and Weil, 2008). Decreased BD and hence increased soil porosity, has probably accounted for improved yield observed at AFS experimental site (Tables 4.71; 4.72; and 4.73).

Simple linear regression analysis for clay loam BD data showed  $R^2$ =0.711 (Table 4.60), and small p-value<0.05 (Table 4.61). The model predicting clay loam soil's BD changes with various pulp application rates is significant. For the 2008 growing season, parameter estimates (Table 4.62) for BD at AFS site indicates that for 1% increase in RRPP application rate a decrease of 0.026 Mg m<sup>-3</sup> in top soil (0.15 m depth) BD is expected.

A second consecutive year of RRPP application to the clay loam did not show significant changes in soil BD (Table 4. 49). Moreover, increases in soil BD were observed when compared with BD means from the previous year. Probably, slightly altered cultivation practices in 2009 are responsible for these results, i.e. cultivation with heavy machinery (no fertilizer was applied in 2008), increased weed pressure on the second year required more frequent tillage operation (mechanical and manual). At the top 0.1 m of cultivated soils, BD is subject to constant changes due to yearly cultivation practices. Therefore, seasonal BD fluctuations are expected for cultivated top soils. Price and Voroney (2007) reported that significant decrease in clay loam BD with residual paper pulp application rates was shown only during the third year of their study.

The same study reported positive response in wet aggregate stability, and increased surface water infiltration.

Simple linear regression analysis for 2009 BD data indicates low R<sup>2</sup> (Table 4.63), and the overall model is not significant (p-value>0.05; Table 4.64). On the second year, the model is inadequate in predicting changes in BD values with changes in pulp application rates.

Ameliorating sandy loam with RRPP in 2008 (1<sup>st</sup> growing season), resulted in statistically significant decrease in BD for 4% v/v pulp application when compared to 1% v/v rate (Table 4.49), but not to control. In contrast to this study, Price and Voroney (2007) reported significant decrease in bulk density with increased paper sludge rates in all the three years of a study in coarse sandy loam. In 2008 (Kolhiko site), mean BD on all experimental plots at the sandy loam were larger than BD at the clay loam. This result is expected since clay loams have more intra-aggregate spaces when compared to sandy soils. Also to recall that in this study the RRPP was applied at a very fine particle size form, in contrast to other studies evaluating similar materials.

Simple linear regression analysis of 2008 BD measurements for the Kolhiko experimental plots resulted in relatively large R<sup>2</sup>=0.668 (Table 4.65). As a result, RRPP application rates account for 66.8% of BD variability observed in clay loam site for 1<sup>st</sup> growing season. Overall model predictability is significant as shown from the small p-value<0.011 (Table 4.66). From parameter estimates table (Table 4.67) we derive that for every 1% increase in RRPP application rates (1<sup>st</sup> dose) we may expect a decrease in sandy loam BD equal to 0.031 Mg m<sup>-3</sup>.

BD data collected in fall 2009 from the Kolhiko experimental area were analyzed with one-way ANOVA (replications were treated as blocks). Comparison of BD means with t-student's test (Table 4.49) indicates statistically significant decrease in BD for plots treated with

the larger pulp application rate (4% v/v). All treatments during the second experimental year revealed an increase in mean BD, a trend which was observed at AFS site, as well. Soil compaction may be related to cultivation practices because the soil was disked and rotary tilled more frequently (at least two-three times before each crop planting to incorporate plant residues into the soil) compared to the previous year. No plowing was done in 2009. Excessive cultivation practices may oxidize soil organic matter, and as a result, may reduce soil mineral particles aggregation. This agrees with Nemati et al (2000b), which reported that ameliorating sandy loam with de-inked and secondary paper sludge mixture deteriorated the physical properties of that soil. They observed a significant decrease in wet aggregate stability. It was assumed that increased soil aeration resulted in increased microbial activity and therefore, induced increased paper sludge degradation and destruction of structure linkages between organic matter, Fe or Al, and mineral particles. In this study, increases in BD may only partially be attributed to pulp application rates because the trend was observed for all treatments (controls included) on both experimental sites.

At the end of the second growing season (Kolhiko site), simple linear regression analysis for BD data collected from sandy loam location indicates an R<sup>2</sup>=0.614 (Table 4.68). The overall model is significant since p-value=0.023 (Table 4.69). From the parameter estimate table we conclude that 1% increase in RRPP application rate should result in 0.041 Mg m<sup>-3</sup> decrease in clay loam BD (Table 4.70). Therefore, the simple liner regression model predicts a 1.32 times larger decrease in BD for sandy loam when the same pulp ratio is added for second consecutive year, compared to unamended plots.

### Zea mays L. saccharata (sweet corn) yield

Results of average sweet corn fresh ear are presented in Table 4.70. These data are the grand average of two sampling dates in each location for 2009 and one sampling date in 2008. The yield mean for each RRPP rate represent the average of sampling dates for ten randomly sampled corn ears from the middle row of each experimental plot and block. The same crop variety (Legend F1 hybrid) was cultivated in both years.

The comprehensive data on sweet corn yield indicated and supported a common trend at each location and year for an increased yield in the mean ear fresh weight with increased application of RRPP material. Although the yield increases were proportional to the application rate, the mean was in all cases statistically significant for the 4% v/v application rate, indicating that this amount of RRPP resulted consistently in yield increases satisfactory for farmers use.

The regression equations predicting yield based on RRPP applications rates for each year and location are as follows:

As shown in the above equations, the initial (soil stored) higher fertility of the AFS site over the Kolhiko location reflects the means comparison results shown in Table 4.70. For example, for no RRPP application the fresh ear weight is always higher at the AFS location than in Kolhiko. Combined equations from two years for each location are shown below and illustrate in general similar trends for the two soil types with the yearly ones.

Kolhiko yield 2009+2008=226.94+12.428\*RRPP rate  $(R^2=0.396, p-value=0.0002)$ 

AFS yield 2009+2008=253.62+15.80\*RRPP rate  $(R^2=0.577, p-value=0.0001)$ 

The 4% v/v rate can be recommended to farmers as a starting rate for any field since results were similar in trend in both soil types and during two years of diverse weather conditions and slightly different cultural practices. The low input of irrigation, fertilization and pest management approach (LISA) used during the first two years of this study, appears to benefit from this material in the case of sweet corn. During the next two years, it is planned (based on the reported results) to further evaluate this material under conventional management practices at each site, to obtain additional information on the behavior of this material in time and under more supporting management practices.

# Lactuca sativa L. (lettuce) yield

The results of lettuce yield (fresh weight of one head) shown in Table 4.71 are for two different lettuce varieties Butterhead type grown in 2008 and Cos type grown in 2009. The values are grand averages from three sampling dates at AFS and one sampling date at Kolhiko. Five lettuce heads were sampled from each experimental plot per block.

Linear regression analysis performed with treatments as numerical and continuous variables resulted in the following equations for each year and location (regression equations were not developed from pooled data from the two years, since different lettuce varieties were used in each year):

Kolhiko 2008 lettuce yield =215.42+20.215\*RRPP rate (R<sup>2</sup>=0.18, p-value=0.1145)

AFS 2008 lettuce yield=517.59+44.345\*RRPP rate (R<sup>2</sup>=0.48, p-value=0.0041)

Kolhiko 2009 lettuce yield=288.91+34.594\*RRPP rate (R<sup>2</sup>=0.55, p-value=0.0015)

AFS 2009 lettuce yield=857.54+134.912\*RRPP rate (R<sup>2</sup>=0.35, p-value=0.0207)

The comprehensive data of lettuce yield from all locations and years shown in Table 4.71 are similar to the results for sweet corn yield and indicated and highly supported a common trend at each location for an increased yield in the mean lettuce fresh weight with increased application of RRPP material. All rates of RRPP applications resulted in numerical yield increases over the control plots. Furthermore the 4% v/v application rate caused statistically significantly higher yield in all cases except for the year 2008 in Kolhiko location, where it resulted in a 28.2% yield increase over the control. The higher application rate (4% v/v) could be recommended to farmers, as a starting rate for any field, since results were similar in trend in both soil types varying largely in their texture and other properties. Furthermore, the trend was shown consistently during two years of diverse weather conditions and slightly different cultural practices. The low input (irrigation, fertilization and pest management) approach used (LISA), appears to benefit from this material in the case of lettuce. For the next two years, this study will continue the evaluation of the RRPP under conventional management practices at each site, to obtain additional information on the behavior of this material in time and under more supporting management practices.

Seasonal yield variation is most possibly related to different lettuce varieties cultivated each year. In addition, yield variation among the two locations, may have been caused by the differences in fertility levels and the slightly different planting dates. However, the author expects that major yield differences resulted from the varying weather conditions in each location, and mainly the air temperature and the associated heat degree days accumulated at each experimental site. For example, the annual average air temperature at the AFS location is warmer by approximately 1.7°C than the Kolhiko location, resulting in higher accumulation of growth heat units. Kolhiko location also exhibited more frost days (<0°C), occurring in both years.

Average weather data for both locations and years are presented in Appendix C (Tables C.1 and C.2; and Figures C.1 to C.4).

## Brassica oleracea L. (cabbage) yield

Table 4.72 presents the results from year 2008 for the fresh cabbage weight. Each value is the grand average of 5 samples per experimental plot and from each block and from two sampling dates at AFS and one sampling date at Kolhiko location. The linear regression equations resulted for each location are:

Kolhiko cabbage yield =760.059+178.004\*RRPP rate ( $R^2$ =0.500, p-value=) AFS cabbage yield=2392.63+165.988\*RRPP rate ( $R^2$ =0.359, p-value=)

Cabbage yield data were not taken in 2009 due to the late crop planting (March 14, 2009). As a result, not enough time existed for full maturation and harvesting before planting of the next crop in the cycle (sweet corn was sown on May 9, 2009). Therefore, cabbage plants were disked over and incorporated in the soil surface by rotary tiling. Results from 2008 shown in Table 4.72 are the means of 3 sampling dates in AFS and two samplings in Kolhiko. Data demonstrate similar yield trends with the other two crop species grown in both locations. The higher application rates of RRPP resulted in higher cabbage yields, with the exception of control plots in Kolhiko, where yield was only higher than the 1% v/v application rate but not statistically significant. In both locations the 4% v/v application rate resulted in significantly higher than the control plots yield.

# Leaf chlorophyll levels for Zea mays L. saccharata (sweet corn)

Chlorophyll relative levels were measured with the Minolta SPAD 502 leaf chlorophyll meter, to investigate possible relationships between leaf chlorophyll status, yield and application rates. This instrument is frequently used for Nitrogen management recommendations in corn and

other crop species (Schepers et al. 1992; Piekielek and Fox, 1992; Varvel et al. 1997; Scharf et al. 2006). SPAD 502 is a simple, user friendly sensor, and if validated appropriately, becomes a powerful management tool. This sensor allows for non-destructive sampling methods and was utilized to complement studies related to remote sensing for in-season site specific N management (Miao et al.2009). The data presented in Table 4.73 are the grand mean values from two sampling dates (close to maturity) in both locations. Three measurements were taken (upper, middle, and lower leaf) per sweet corn plant, from five plants randomly selected per experimental plot.

A significant difference in chlorophyll level was shown for the higher application rate (4% v/v). SPAD units increased proportionally to RRPP application rates as shown in the regression equations given bellow. The regression equation developed between SPAD and the four RRPP application rates were highly significant and the equations for each location are:

Kolhiko SPAD units=
$$48.95+0.3857*RRPP$$
 application rate ( $R^2=992$ ,  $p$ -value= $0.0041$ )  
AFS SPAD units= $47.45+1.064*RRPP$  application rate ( $R^2=0.951$ ,  $p$ -value= $0.0248$ ))

Regression analysis of mean SPAD and mean fresh weight results, demonstrated a high correlation between yield (mean fresh ear weight) and SPAD units (R<sup>2</sup>=0.739 and 0.878 for AFS and Kolhiko locations, respectively), although marginally not statistically significant. The regression equations predicting mean ear fresh weight for each location based on SPAD measurements are:

Kolhiko yield=1194.87+29.105\*SPAD units (
$$R^2$$
=0.879, p-value=0.0623)  
AFS yield=48.295+4.905\*SPAD units ( $R^2$ =0.739, p-value=0.1401)

The above equations can be used for yield prediction on each location and for various levels of RRPP application rates, within the range applied in this study and for each soil type.

The SPAD unit equations can be used for mean chlorophyll level prediction on each location and for various levels of RRPP application rates, within the range applied in this study and for each soil. A combination of both equations predicting SPAD and yield may be a useful management tool for this crop species (sweet corn) grown at each location, and will be further validated in the next phase of this study.

# Leaf chlorophyll levels for Lactuca sativa L. (lettuce)

The data presented in Table 4.74 are the grand mean values from one sampling date (close to maturity) in both locations for lettuce crops. Three measurements were taken (upper, middle, and lower leaf) per plant, from five plants randomly selected per experimental plot. The results showed significant difference for the AFS location but not for the Kolhiko location, although in the latter case SPAD units proportionally increased with increasing rates of RRPP. Pulp application at 4% v/v rates on clay loam resulted in a statistically significant increase in mean chlorophyll level in lettuce leaves. This result agrees with the significant increases in lettuce fresh weight yield observed for both locations on 2009 for the larger (4% v/v) RRPP amendment.

The regression equations between SPAD and the four RRPP application rates were highly significant and the individual equations for each location are:

Kolhiko SPAD units=24.48+0.5763\*RRPP application rate ( $R^2=0.946$ , p-value=0.02476)

AFS SPAD units=38.24+1.841\*RRPP application rate ( $R^2=0.973$ , p-value=0.0136)

The above equations reflect the higher potential productivity for the AFS field even for 0 RRPP application rate.

The equations predicting mean lettuce head fresh weight for each location based on SPAD measurements are:

Kolhiko yield = 
$$-1245.53 + 62.962 * SPAD$$
 units ( $R^2$ =0.942,  $p$ -value=0.0295)  
AFS yield =  $-1085.06 + 51.702 * SPAD$  units ( $R^2$ =0.909,  $p$ -value=0.0463)

During the second growing season, a high correlation for fresh lettuce yield and measured SPAD units was observed at both locations. Relative chlorophyll level, and therefore lettuce nitrogen assimilation seems to be significantly related to pulp application rates at both sites.

The SPAD units were equally well correlated with yield and RRPP application rates for both crop species (sweet corn and lettuce) examined at both locations, indicating that this agronomic characteristic may assist in developing optimum yield management practices.

Soil Volumetric Water Content (VWC) and Apparent Electrical Conductivity (ECa)

# measured with the WET sensor

Various data sets were taken throughout the two years with the WET sensor, to monitor surface soil volumetric water content (VWC) and the corresponding apparent electrical conductivity (ECa) at the same time. Table 4.75 presents representative sets of VWC from both locations and years. Data for ECa are shown in Table 4.76.

In general, the results of VWC and ECa were not consistent in both years and locations; this is due to measurements taken at different times of the year and at variable moisture conditions. It appears, that ECa was not affected by RRPP rates in all but one case (Kolhiko 2009) where the zero RRPP rate had significantly less ECa than the other rates. The RRPP material did not increase this value above critical limits. All values were below threshold values (<2 dS m<sup>-1</sup>) limiting plant growth and yield.

The VWC was the most inconsistent changing among soil types and making it difficult to measure reliable results and form proper conclusions. It appears that increasing RRPP rates did not increase VWC; in contrast many sampling sets indicated the opposite. The control plots in

sandy loam soil exhibited higher VWC than the treated plots. The same trend was shown in additional data sets (taken but not presented in this study).

In general, analysis of seasonal variation of VWC mainly and less ECa, deserves more carefully examination and it also appears that the "blocks" effects influenced the treatment effects. Also, cultivation practices concentrated on the top soil (0-0.15 m depth) may have affected the seasonal variation of VWC.

Table 4.1 Mean SOC for RRPP application rates in all locations and years.

Tuestment	SOC	SOC	SOC	SOC
Treatment	(Kolhiko 2008)	(Kolhiko 2009)	(AFS 2008)	(AFS 2009)
RRPP % (v/v)±	%	%	%	%
4	1.106a†	1.321a†	1.287a†	1.658a†
2	1.165a	1.17ab	1.273a	1.609a
1	0.958ab	1.108ab	1.273a	1.44b
0 (Control)	0.782b	0.99b	1.246a	1.343b

Notes: †Values followed by the same letter are not statistically significant at p-value<0.05 (Student's t-test).

 $\pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m.

RRPP=Recycled residual Paper Pulp; SOC=Soil Organic Carbon, AFS=American Farm School.

Table 4.2 Summary of fit for SOC at Kolhiko 2008 vs. treatments.

$\mathbb{R}^2$	$\mathbb{R}^2$ Adj	RMSE	MR	Observations
0.448	0.247	0.21	1.002	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response.

Table 4.3 Analysis of variance for SOC at Kolhiko 2008 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.394	0.098	2.228	0.1324
Error	11	0.486	0.044		
C. Total	15	0.880			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square.

Table 4.4 Parameter estimates for SOC at Kolhiko 2008 vs. treatments.

Term	Estimate	SE	t Ratio	Prob> t
Intercept	0.864	0.0814	10.61	<.0001*
Treatments	0.0792	0.0355	2.23	0.0476*
Rep-Block[1]	-0.1215	0.0910	-1.33	0.2089
Rep-Block[2]	0.162	0.0910	1.78	0.1027
Rep-Block[3]	0.009	0.0910	0.10	0.9230

Notes: Values indicated with (\*) are statistically significant at level of significance  $\alpha$ =0.05.

The model was run as Simple Regression with replications used as blocks.

SE=Standard Error.

Table 4.5 Summary of fit for SOC at Kolhiko 2009 vs. treatments.

$\mathbb{R}^2$	$R^2Adj$	RMSE	MR	Observations
0.506	0.326	0.18	1.147	16

RMSE=Root Mean Square Error; MR= Mean of Response.

Table 4.6 Analysis of variance for SOC at Kolhiko 2009 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.363	0.091	2.813	0.078
Error	11	0.355	0.032		
C. Total	15	0.717			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square.

Table 4.7 Summary of fit for SOC at AFS 2008 vs. treatments.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.204	-0.09	0.121	1.27	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table 4.8 Analysis of variance for SOC at AFS 2008 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.0414	0.0103	0.7045	0.6052
Error	11	0.1616	0.0146	0.6052	
C. Total	15	0.203			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table 4.9 Summary of fit for SOC at AFS 2009 vs. treatments.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.845	0.789	0.081	1.513	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table 4.10 Analysis of variance for SOC at AFS 2009 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.390	0.098	14.9904	0.0002
Error	11	0.072	0.007		
C. Total	15	0.462			

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table 4.11 Parameter estimates for SOC at AFS 2009 vs. treatments.

Term	Estimate	SE	t Ratio	Prob> t
Intercept	1.372	0.031	43.908	0.0000*
Treatments	0.080	0.014	5.887	0.0001*
Rep-Block[1]	-0.041	0.035	-1.175	0.2647
Rep-Block[2]	-0.140	0.035	-4.009	0.0021*
Rep-Block[3]	0.132	0.035	3.784	0.0030*

Note: Values indicated with (\*) are statistically significant at level of significance  $\alpha$ =0.05.

The model was run as Simple Regression with replications used as blocks.

SE=Standard Error; AFS=American Farm School.

Table 4.12 Mean pH for RRPP application rates in all years and locations.

Treatment	pН	pН	pН	pН
Treatment	(AFS 2008)	(AFS 2009)	(Kolhiko 2008)	(Kolhiko 2009)
RRPP% (v/v)±	mol H <sup>+</sup> L <sup>-</sup>			
4	7.450a†	7.270a†	6.900a†	7.270a†
2	7.338a	7.268a	7.000a	7.295a
1	7.225a	7.243a	6.925a	7.168a
0 (Control)	7.413a	7.233a	6.463b	6.653b

Notes:  $\dagger$ Values at the same column followed by the same letter are not statistically significant at p<0.05 (Student's t-test).

 $\pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15 m.

RRPP=Recycled residual Paper Pulp; SOC=Soil Organic Carbon; AFS=American Farm School.

Table 4.13 Summary of fit for pH at AFS 2008 vs. treatments.

$\mathbb{R}^2$	R <sup>2</sup> Adj	RMSE	MR	Observations
0.492	0.307	0.159	7.356	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error, MR= Mean of Response; AFS=American Farm School.

Table 4.14 Analysis of variance for pH at AFS 2008 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.270	0.068	2.659	0.090
Error	11	0.279	0.025		
C. Total	15	0.549			

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table 4.15 Summary of fit for pH at AFS 2009 vs. treatments.

$\mathbb{R}^2$	$R^2Adj$	RMSE	MR	Observations
0.097	-0.231	0.075	7.253	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table 4.16 Analysis of variance for pH at AFS 2009 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.007	0.002	0.295	0.875
Error	11	0.063	0.006		
C. Total	15	0.069			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table 4.17 Summary of fit for pH at Kolhiko2008 vs. treatments.

$\mathbb{R}^2$	$R^2Adj$	RMSE	MR	Observations
0.523	0.350	0.287	6.822	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response.

Table 4.18 Analysis of variance for pH at AKolhiko2008 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.997	0.249	3.017	0.066
Error	11	0.908	0.083		
C. Total	15	1.905			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table 4.19 Summary of fit for pH at Kolhiko 2009 vs. treatments.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.500	0.3191	0.242	7.096	16

RMSE=Root Mean Square Error; MR= Mean of Response.

Table 4.20 Analysis of variance for pH at Kolhiko 2009 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.647	0.162	2.754	0.083
Error	11	0.646	0.059		
C. Total	15	1.293			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square.

Table 4.21 Mean ECs for RRPP application rates in all locations and years.

Tuestment	EC	EC	EC	EC
Treatment	(AFS 2008)	(AFS 2009)	(Kolhiko 2008)	(Kolhiko2009)
RRPP% (v/v)±	dS m <sup>-1</sup>	dS m <sup>-1</sup>	dS m <sup>-1</sup>	dS m <sup>-1</sup>
4	0.759a†	1.534a†	0.263a†	0.651bc†
2	0.810a	1.3612a	0.268a	0.813ab
1	0.813a	1.367a	0.333a	0.888a
0	0.741a	1.599a	0.265a	0.443c

Notes: † Values at the same column followed by the same letter are not statistically significant at p<0.05 (Student's t-test).

 $\pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15 m.

RRPP=Recycled residual Paper Pulp; SOC=Soil Organic Carbon; AFS=American Farm School.

Table 4.22 Summary of fit for ECs at AFS 2008 vs. treatments.

$\mathbb{R}^2$	$\mathbb{R}^2 \mathbf{Adj}$	RMSE	MR	Observations
0.454	0.256	0.1182	0.781	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table 4.23 Analysis of variance for ECs at AFS 2008 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.127	0.032	2.289	0.125
Error	11	0.153	0.014		
C. Total	15	0.280			

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table 4.24 Summary of fit for ECs at AFS 2009 vs. treatments.

$\mathbb{R}^2$	$R^2Adj$	RMSE	MR	Observations
0.345	0.107	0.283	1.465	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table 4.25 Analysis of variance for ECs at AFS 2009 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.463	0.116	1.450	0.282
Error	11	0.878	0.080		
C. Total	15	1.341			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table 4.26 Summary of fit for ECs at Kolhiko 2008 vs. treatments.

$\mathbb{R}^2$	$R^2Adj$	RMSE	MR	Observations
0.130	-0.187	0.078	0.282	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response.

Table 4.27 Summary of fit for ECs at Kolhiko 2009 vs. treatments.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.345	0.107	0.283	1.465	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response.

Table 4.28 Analysis of variance for ECs at Kolhiko 2009 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.074	0.018	0.323	0.857
Error	11	0.626	0.057		
C. Total	15	0.699			

SS=Sum of Squares; MS= Mean Square.

Table 4.29 Mean Total N% for RRPP in all locations and years.

Treatment	Total N (AFS 2008)	Total N (AFS 2009)	Total N (Kolhiko 2008)	Total N (Kolhiko 2009)
-RRPP% (v/v)±-	%	%	%	%
4	0.165a†	0.203a†	0.093a†	0.121a†
2	0.170a	0.203a	0.086a	0.111a
1	0.163a	0.189a	0.098a	0.134a
0 (Control)	0.156a	0.191a	0.088a	0.112a

Notes:  $\dagger$ Values at the same column followed by the same letter are not statistically significant at p<0.05 (Student's t-test).

 $\pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15 m.

RRPP=Recycled residual Paper Pulp; SOC=Soil Organic Carbon; AFS=American Farm School.

Table 4.30 Summary of fit for Total N% at AFS 2008 vs. treatments.

$\mathbb{R}^2$	$R^2Adj$	RMSE	MR	Observations
0.116	-0.205	0.015	0.164	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table 4.31 Analysis of variance for Total N% AFS 2008 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.000	0.000	0.361	0.831
Error	11	0.003	0.000		
C. Total	15	0.003			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table 4.32 Summary of fit for Total N% at AFS 2009 vs. treatments.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.300	0.045	0.018	0.196	16

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table 4.33 Analysis of variance for Total N% AFS 2009 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.001	0.000	1.178	0.373
Error	11	0.003	0.000		
C. Total	15	0.005			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table 4.34 Summary of fit for Total N% at Kolhiko 2008 vs. treatments.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.125	-0.194	0.014	0.091	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response.

Table 4.35 Analysis of variance for Total N% Kolhiko 2008 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.000	0.000	0.391	0.811
Error	11	0.002	0.000		
C. Total	15	0.002			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square.

Table 4.36 Summary of fit for Total N% at Kolhiko 2009 vs. treatments.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.300	0.045	0.018	0.196	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response.

Table 4.37 Analysis of variance for Total N% Kolhiko 2009 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.001	0.000	1.178	0.373
Error	11	0.003	0.000		
C. Total	15	0.005			

SS=Sum of Squares; MS= Mean Square.

Table 4.38 Mean comparison of C/N for RRPP application rates in all locations and years.

Treatment	C/N (AFS 2008)	C/N (AFS 2009)	C/N (Kolhiko 2008)	C/N (Kolhiko 2009)
-RRPP% $(v/v)\pm$ -				
4	8.45a†	8.000a†	10.250a†	11.300a†
2	8.4a	8.225a	10.625a	11.125a
1	8.55a	8.150a	10.125a	10.300a
0 (Control)	8.55a	7.075a	9.750a	9.525a

Notes: † Values at the same column followed by the same letter are not statistically significant at p<0.05 (Student's t-test).

 $\pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15 m.

RRPP=Recycled residual Paper Pulp; SOC=Soil Organic Carbon; AFS=American Farm School.

Table 4.39 Summary of fit for C/N at AFS 2008 vs. treatments.

$\mathbb{R}^2$	$R^2Adj$	RMSE	MR	Observations
0.157	-0.149	0.616	8.488	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table 4.40 Analysis of variance for C/N AFS 2008 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.779	0.195	0.513	0.728
Error	11	4.178	0.380		
C. Total	15	4.957			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table 4.41 Summary of fit for C/N at AFS 2009 vs. treatments.

$\mathbb{R}^2$	$R^2Adj$	RMSE	MR	Observations
0.455	0.256	1.073	7.863	16

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table 4.42 Analysis of variance for C/N AFS 2009 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	10.544	2.636	2.291	0.125
Error	11	12.654	1.150		
C. Total	15	23.198			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table 4.43 Summary of fit for C/N at Kolhiko 2008 vs. treatments.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.731	0.633	0.598	10.188	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response.

Table 4.44 Analysis of variance for C/N Kolhiko 2008 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	10.669	2.667	7.467	0.004
Error	11	3.929	0.357		
C. Total	15	14.598			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square.

Table 4.45 Parameter estimates for C/N Kolhiko 2008 vs. treatments.

Term	Estimate	SE	t Ratio	Prob> t
Intercept	9.975	0.231	43.095	0.0000*
Treatments	0.121	0.101	1.202	0.2546
Rep-Block[1]	0.937	0.259	3.623	0.0040*
Rep-Block[2]	0.413	0.259	1.594	0.1392
Rep-Block[3]	-0.137	0.259	-0.531	0.6058

Note: Values indicated with (\*) are statistically significant at level of significance  $\alpha$ =0.05.

The model was run as Simple Regression with replications used as blocks.

SE=Standard Error.

Table 4.46 Summary of fit for C/N at Kolhiko 2009 vs. treatments.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.285	0.026	2.385	10.563	16

RMSE=Root Mean Square Error; MR= Mean of Response.

Table 4.47 Analysis of variance for C/N at Kolhiko 2009 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	24.997	6.249	1.099	0.405
Error	11	62.560	5.687		
C. Total	15	87.558			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square.

Table 4.48 Mean SOC for RRPP application rates in all locations and years. Sample analyses at the University of Belgrade, Serbia.

Treatment	SOC (AFS 2008)	SOC (AFS 2009)	SOC (Kolhiko 2008)	SOC (Kolhiko 2009)
-RRPP% (v/v)±-	%	%	%	%
4	1.390a†	1.633a†	0.958a†	1.323a†
2	1.420a	1.655a	0.908a	1.218a
1	1.393a	1.553a	0.983a	1.378a
0 (Control)	1.343a	1.355a	0.853a	1.068a

Notes: † Values followed by the same letter are not statistically significant at p-value<0.05 (Student's t-test).

 $\pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m.

RRPP=Recycled residual Paper Pulp; SOC=Soil Organic Carbon; AFS=American Farm School.

Table 4.49 Summary of fit for SOC at AFS 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.065	-0.274	0.078	1.386	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table 4.50 Analysis of variance for SOC at AFS 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.005	0.001	0.193	0.937
Error	11	0.067	0.006		
C. Total	15	0.072			

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table 4.51 Summary of fit for SOC at AFS 2009 vs. treatments. Analyses at the University of Belgrade, Serbia.

$\mathbb{R}^2$	$R^2Adj$	RMSE	MR	Observations
0.643	0.513	0.200	1.549	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; .AFS=American Farm School.

Table 4.52 Analysis of variance for SOC at AFS 2009 vs. treatments. Analyses at the University of Belgrade, Serbia.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.793	0.198	4.944	0.016
Error	11	0.441	0.040		
C. Total	15	1.234			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table 4.53 Parameter estimates for SOC at AFS 2009 vs. treatments. Analyses at the University of Belgrade, Serbia.

Term	Estimate	SE	t Ratio	Prob> t
Intercept	1.439	0.078	18.546	0.000*
Treatments	0.063	0.034	1.861	0.090
Rep-Block[1]	-0.086	0.087	-0.995	0.341
Rep-Block[2]	-0.266	0.087	-3.070	0.011*
Rep-Block[3]	0.284	0.087	3.272	0.007*

Note: Values indicated with (\*) are statistically significant at level of significance  $\alpha$ =0.05.

The model was run as Simple Regression with replications used as blocks.

SE=Standard Error.

Table 4.54 Summary of fit for SOC at Kolhiko 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

${f R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.083	-0.251	0.158	0.925	16

RMSE=Root Mean Square Error; MR= Mean of Response.

Table 4.55 Analysis of variance for SOC at Kolhiko 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.025	0.006	0.248	0.905
Error	11	0.274	0.025		
C. Total	15	0.299			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square.

Table 4.56 Summary of fit for SOC at Kolhiko 2009 vs. treatments. Analyses at the University of Belgrade, Serbia.

$\mathbb{R}^2$	$\mathbb{R}^2 \mathbf{Adj}$	RMSE	MR	Observations
0.219	-0.064	0.350	1.246	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response.

Table 4.57 Analysis of variance for SOC at Kolhiko 2009 vs. treatments. Analyses at the University of Belgrade, Serbia.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.379	0.095	0.773	0.565
Error	11	1.348	0.123		
C. Total	15	1.727			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square.

Table 4.58 Mean	BD for RRP	Papplication r	rates in both	locations and	vears.
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Tuestment	BD	BD	BD	BD
Treatment	(AFS 2008)	(AFS 2009)	(Kolhiko 2008)	(Kolhiko 2009)
RRPP% (v/v)±	Mg m <sup>-3</sup>	Mg m <sup>-3</sup>	Mg m <sup>-3</sup>	Mg m <sup>-3</sup>
4	1.098b†	1.245a†	1.108b†	1.300b†
2	1.177ab	1.253a	1.218ab	1.493a
1	1.194a	1.223a	1.268a	1.465a
0	1.198a	1.230a	1.210ab	1.460a

Notes: † Values followed by the same letter are not statistically significant at p-value<0.05 (Student's t-test).

 $\pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m.

RRPP=Recycled residual Paper Pulp; BD= Bulk Density; AFS=American Farm School.

Table 4.59 Summary of fit for BD at AFS 2008 vs. treatments.

$\mathbb{R}^2$	$R^2Adj$	RMSE	MR	Observations
0.711	0.606	0.049	1.166	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; BD=Bulk Density; AFS=American Farm School.

Table 4.60 Analysis of variance for BD at AFS 2008 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.064	0.016	6.774	0.005*
Error	11	0.026	0.002		
C. Total	15	0.090			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; BD=Bulk Density; AFS=American Farm School.

Values with (\*) are statistically significant (p<0.05).

Table 4.61 Parameter estimates for BD at AFS 2008 vs. treatments.

<b>Term</b>	<b>Estimate</b>	SE	t Ratio	Prob> t
Intercept	1.212	0.019	64.245	0.000*
Treatments	-0.026	0.008	-3.161	0.009*
Rep-Block[1]	-0.052	0.021	-2.479	0.031*
Rep-Block[2]	0.039	0.021	1.867	0.089
Rep-Block[3]	-0.047	0.021	-2.242	0.047*

Note: Values indicated with (\*) are statistically significant at level of significance  $\alpha$ =0.05.

The model was run as Simple Regression with replications used as blocks.

SE=Standard Error; BD=Bulk Density; AFS=American Farm School.

Values with (\*) are statistically significant (p<0.05).

Table 4.62 Summary of fit for BD at AFS 2009 vs. treatments.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.299	0.045	0.042	1.238	16

RMSE=Root Mean Square Error; MR= Mean of Response; BD=Bulk Density; AFS=American Farm School.

Table 4.63. Analysis of variance for BD at AFS 2009 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.008	0.002	1.176	0.374
Error	11	0.019	0.002		
C. Total	15	0.027			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; BD=Bulk Density; AFS=American Farm School.

Table 4.64 Summary of fit for BD at Kolhiko 2008 vs. treatments.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.668	0.548	0.078	1.201	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; BD=Bulk Density.

Table 4.65 Analysis of variance for BD at Kolhiko 2008 vs. treatments.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.134	0.034	5.543	0.011*
Error	11	0.067	0.006		
C. Total	15	0.201			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; BD=Bulk Density.

Values with (\*) are statistically significant (p<0.05).

Table 4.66 Parameter estimates for BD at Kolhiko 2008 vs. treatments.

Term	Estimate	SE	t Ratio	Prob> t
Intercept	1.255	0.030	41.618	0.000*
Treatments	-0.031	0.013	-2.361	0.038*
Rep-Block[1]	-0.076	0.034	-2.243	0.046*
Rep-Block[2]	0.109	0.034	3.244	0.008*
Rep-Block[3]	-0.076	0.034	-2.243	0.046*

Note: Values indicated with (\*) are statistically significant at level of significance  $\alpha$ =0.05.

The model was run as Simple Regression with replications used as blocks.

SE=Standard Error; BD=Bulk Density.

Values with (\*) are statistically significant (p<0.05).

Table 4.67 Summary of fit for BD at Kolhiko 2009 vs. treatments.

$\mathbb{R}^2$	$\mathbb{R}^2 \mathbf{Adj}$	RMSE	MR	Observations
0.614	0.473	0.075	1.429	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; BD=Bulk Density.

Table 4.68 Analysis of variance for BD at Kolhiko 2009 vs. treatments.

	J				
Source	DF	SS	MS	F Ratio	p-value
Model	4	0.098	0.025	4.370	0.023*
Error	11	0.062	0.006		
C. Total	15	0.160			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; BD=Bulk Density.

Values with (\*) are statistically significant (p<0.05).

Table 4.69 Parameter estimates for BD at Kolhiko 2009 vs. treatments.

Term	<b>Estimate</b>	SE	t Ratio	Prob> t
Intercept	1.501	0.029	51.642	0.000*
Treatments	-0.041	0.013	-3.205	0.008*
Rep-Block[1]	0.068	0.032	2.097	0.060
Rep-Block[2]	-0.032	0.032	-0.981	0.348
Rep-Block[3]	0.026	0.032	0.789	0.447

Note: Values indicated with (\*) are statistically significant at level of significance  $\alpha$ =0.05.

The model was run as Simple Regression with replications used as blocks.

SE=Standard Error; BD=Bulk Density.

Values with (\*) are statistically significant (p<0.05).

Table 4.70 Mean sweet corn	yield for RRPP application rates	in all locations and years.
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Treatment	Sweet corn¥ fresh weight (AFS 2009)	Sweet corn fresh weight (Kolhiko 2009)	Sweet corn fresh weight (AFS 2008)	Sweet corn fresh weight (Kolhiko 2008)
RRPP% (v/v)±	g/ear	g/ear	g/ear	g/ear
4	306.0a†	272.3a†	323.1a†	252.0a†
2	286.5b	255.5ab	295.9b	249.7a
1	285.3b	247.0ab	248.0c	243.4ab
0 (Control)	283.0b	223.0b	232.3c	219.2b

Notes: †Values followed by the same letter are not statistically significant at p-value<0.05 (Student's t-test).

RRPP=Recycled residual Paper Pulp; AFS=American Farm School.

Table 4.71 Mean lettuce fresh yield for RRPP application rates in all locations and years.

Treatment	Lettuce fresh weight (AFS 2008)	Lettuce fresh weight (AFS 2009)	Lettuce fresh weight (Kolhiko 2008)	Lettuce fresh weight (Kolhiko 2009)
RRPP% (v/v)±	g/head	g/head	g/head	g/head
4	701.8a†	1301.5a†	291.5a†	422.5.3a†
2	584.8ab	1029.5ab	282.3a	396.3.5ab
1	579.8ab	1010.8ab	257.0a	344.8ab
0	568.3b	892.8b	227.5a	273.5b

Notes: †Values followed by the same letter are not statistically significant at p-value<0.05 (Student's t-test).

± (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15 m. RRPP=Recycled residual Paper Pulp; AFS=American Farm School.

 $<sup>\</sup>pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m.

<sup>¥ 10</sup> fresh sweet corn ear weights were measured/plot/sampling date.

Two sampling dates for 2009, one sampling date for 2008.

Table 4.72 Mean cabbage fresh	vield for RRPP application	rates in all locations in 2008.
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Treatment	Cabbage fresh weight (AFS 2008)	Cabbage fresh weight (Kolhiko 2008)
RRPP% (v/v)±	g	g
4	3003.5a†	1427.8a†
2	2646.3ab	1243.7ab
1	2633.8ab	860.1c
0	2302.0c	909.3bc

Notes: †Values followed by the same letter are not statistically significant at p-value<0.05 (Student's t-test).

RRPP=Recycled residual Paper Pulp; AFS=American Farm School.

No cabbage yield data were collected in 2009.

Table 4.73 Mean chlorophyll level on sweet corn leaves for RRPP application rates in both locations in 2009.

Treatment	Chlorophyll level on sweet corn leaves (AFS 2009)	Chlorophyll level on sweet corn leaves (Kolhiko 2009)
RRPP% (v/v)±	SPAD units	SPAD units
4	51.1a†	50.5a†
2	49.8ab	49.7ab
1	49.0ab	49.3ab
0	47.0b	49.0b

Notes: †Values followed by the same letter are not statistically significant at p-value<0.05 (Student's t-test).

 $<sup>\</sup>pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m.

 $<sup>\</sup>pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m. RRPP=Recycled residual Paper Pulp; AFS=American Farm School.

Table 4.74 Mean chlorophyll level on lettuce leaves for RRPP application rates in both locations in 2009.

Treatment	Chlorophyll level on lettuce leaves (AFS 2009)	Chlorophyll level on lettuce leaves (Kolhiko 2009)
RRPP % (v/v)±	SPAD units	SPAD units
4	45.7a†	26.6a†
2	42.1ab	26.0a
1	39.3b	25.0a
0	38.8b	24.4a

Notes:  $\dagger$ Values followed by the same letter are not statistically significant at p-value < 0.05 (Student's t-test).

RRPP = Recycled residual Paper Pulp; AFS = American Farm School.

Table 4.75 Mean soil surface Volumetric Water Content for RRPP application rates in all locations and years.

	VWC	VWC	VWC	VWC
<b>Treatment</b>	AFS	AFS	Kolhiko	Kolhiko
	22-8-2008	4-3-2009	22-8-2008	17-1- 2009
RRPP % (v/v)±-	\% $m^3/m^{-3}$	\% $m^3/m^{-3}$	\% $m^3/m^{-3}$	\% $m^3/m^{-3}$
4	23.51a†	20.50a†	25.13b†	24.94a†
2	14.12b	17.72b	27.76ab	25.96a
1	16.73ab	20.87a	29.72 a	28.02a
0	16.65ab	21.15a	28.63ab	27.84a

Notes:  $\dagger$ Values followed by the same letter are not statistically significant at p-value < 0.05 (Student's t-test).

 $\pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15 m.

RRPP = Recycled residual Paper Pulp; AFS = American Farm School; VWC=Volumetric Water Content.

 $<sup>\</sup>pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m.

Table 4.76 Mean soil surface Apparent Electrical conductivity for RRPP application rates in all locations and years.

Treatment	ECa AFS 22-8-2008	ECa AFS 4-3-2009	ECa Kolhiko 22-8- 2008	EC Kolhiko 17-1-2009
RRPP % (v/v)±	dS m <sup>-1</sup>	dS m <sup>-1</sup>	dS m <sup>-1</sup>	dS m <sup>-1</sup>
4	1.73a†	1.82a†	1.53a†	0.87a†
2	1.31a	1.88a	1.26a	0.83ab
1	1.39a	1.87a	1.29a	0.82a
0	1.56a	1.89a	1.25a	0.76b

Notes:  $\dagger$ Values followed by the same letter are not statistically significant at p-value < 0.05 (Student's t-test).

 $<sup>\</sup>pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15 m. RRPP = Recycled residual Paper Pulp; AFS = American Farm School; ECa= Apparent Electrical conductivity.

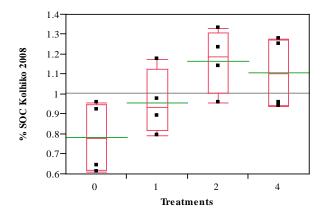


Figure 4.1 Changes in SOC resulting from RRPP application rates at Kolhiko site (0=Control-no RRPP; 1=1% RRPP applied; 2=2% RRPP applied; and 4=4% RRPP applied) at the end of 1<sup>st</sup> growing season (2008). Interior solid lines (red color) are medians, while green horizontal lines indicate means for each treatment. Gray horizontal line is the grand mean.

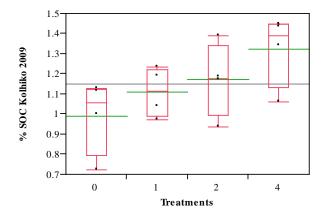


Figure 4.2 Changes in SOC resulting from RRPP application rates at Kolhiko site (0=Control-no RRPP; 1=1% RRPP applied; 2=2%RRPP applied; and 4=4%RRPP applied) at the end of 2<sup>nd</sup> growing season (2009). Interior solid lines (red color) are medians, while green horizontal lines indicate means for each treatment. Gray horizontal line is the grand mean.

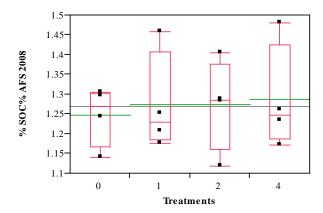


Figure 4.3 Changes in SOC resulting from RRPP application rates at AFS site (0=Control-no RRPP; 1=1% RRPP applied; 2=2% RRPP applied; and 4=4% RRPP applied) at the end of 1<sup>st</sup> growing season (2008). Interior solid lines (red color) are medians, while green horizontal lines indicate means for each treatment. Gray horizontal line is the grand mean.

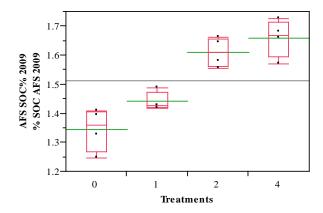


Figure 4.4 Changes in SOC resulting from RRPP application rates at AFS site (0=Control-no RRPP; 1=1% RRPP applied; 2=2% RRPP applied; and 4=4% RRPP applied) at the end of 2<sup>nd</sup> growing season (2009). Interior solid lines (red color) are medians, while green horizontal lines indicate means for each treatment. Gray horizontal line represents grand mean.

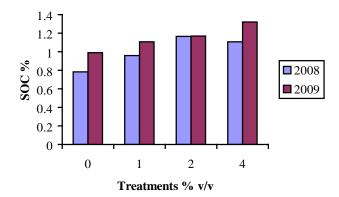


Figure 4.5 SOC comparison at Kolhiko site at the end of 1<sup>st</sup> and 2<sup>nd</sup> growing season.

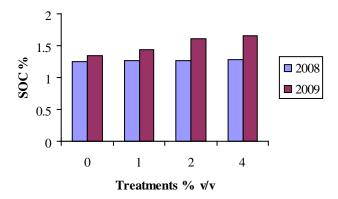


Figure 4.6 SOC comparison at AFS site at the end of 1<sup>st</sup> and 2<sup>nd</sup> growing season.

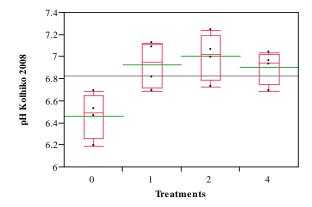


Figure 4.7 Soil pH vs. RRPP application rates at Kolhiko site (0=Control-no RRPP; 1=1% RRPP applied; 2=2% RRPP applied; and 4=4% RRPP applied) at the end of 1<sup>st</sup> growing season (2008). Interior solid lines (red color) are medians, while green horizontal lines indicate means for each treatment. Gray horizontal line is the grand mean.

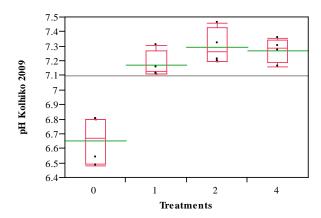


Figure 4.8 Soil oil pH vs. RRPP application rates at Kolhiko site (0=Control-no RRPP; 1=1% RRPP applied; 2=2%RRPP applied; and 4=4%RRPP applied) at the end of 2<sup>nd</sup> growing season (2009). Interior solid lines (red color) are medians, while green horizontal lines indicate means for each treatment. Gray horizontal line is the grand mean.

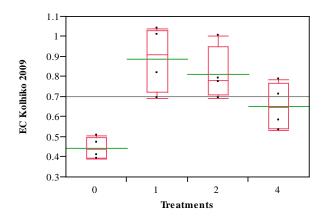


Figure 4.9 Soil EC vs. RRPP application rates at Kolhiko site (0=Control-no RRPP; 1=1% RRPP applied; 2=2% RRPP applied; and 4=4% RRPP applied) at the end of 2<sup>nd</sup> growing season (2009). Interior solid lines (red color) are medians, while green horizontal lines indicate means for each treatment. Gray horizontal line is the grand mean.

# **CHAPTER 5**

# CONCLUSIONS AND RECOMMENDATIONS

# **Conclusions**

With the objective of evaluating recycled residual paper pulp (RRPP) as a soil ameliorating material, two soils, a sandy loam (at Kolhiko, Greece) and a clay loam (at AFS, Thermi, Greece), were treated with RRPP for two consecutive years at 0, 1, 2, and 4% v/v application rates (corresponding to 0, 9.6, 19.2, and 38.4 Mg ha<sup>-1</sup>) incorporated into the upper 0.15 m. The two soils are representative Greek soils, and differed mainly in texture but also in soil fertility. A number of soil physical and chemical properties were studied along with yield measurements and leaf relative chlorophyll levels on three crop species: sweet corn (*Zea mays* L. *saccharata*), cabbage (*Brassica oleracea* L.), and lettuce (*Lactuca sativa* L.).

There are some unique aspects of this study to be considered along with the conclusions. First, all crops were grown and managed under Low Input Sustainable Agriculture (LISA) approach, using reduced water for irrigation, very minimal fertilization and almost zero chemical protection inputs as compared to conventional farming practices. Second, the finely pulverized form of the applied material, as compared to the consolidated form utilized in all other studies reported, is another distinguishing characteristic of this study. Third, the amount of material applied is in the very low range of reported application rates, with the exception of the 4% v/v rate. The above features distinguish this study from all works reported so far with similar materials, and therefore, should be considered in the interpretation of conclusions for land application use.

First-year applications resulted in statistically significant increases in SOC at the sandy loam site for plots treated with 2 and 4% rates. No significant increases were measured at the clay loam site for first-year applications. After two yearly applications, a significant increase in SOC was found with the 4% rate at the sandy loam site. At the clay loam site, significant increases in SOC were obtained with the 2 and 4% application rates only in the second year application.

Soil pH analysis for the sandy loam site indicated a statistically significant increase, although the pH did not change dramatically from ranges suitable for most cultivated crops. In the clay loam site there were no significant changes in soil pH. These results suggest that applying RPPP to a calcareous clay loam will not affect pH levels; at least, after two consecutive doses at this study's rates. It seems that the pulp is more beneficial as an amending material to lightly acidic soils which need pH improvement. Based on our results and the alkaline reaction of the RRPP we can reasonably extrapolate and conclude that the more acidic a soil is, the more effective this material would be in raising soil pH. Studies of Nunes et al. (2008) and Basu et al. (2007) also supported the potential use of paper pulp sludge as a liming agent.

Ameliorating the clay loam with RRPP did not significantly affect soil ECs in either of the two years. After two years of consecutive RRPP addition, significant increases in ECs were detected at the 1 and 2% application rates. These results were expected since pulp analyses from independent laboratories indicated consistently low ECs (Table B.3).

Total Nitrogen was not affected in any of the soils or years. In addition, in both soils, the soil's C/N ratio was not affected by RRPP application rates.

Addition of 4% RRPP to the sandy loam resulted in a significant decrease in bulk density by the end of the second year. In the clay loam, addition of 4% RRPP decreased bulk density

significantly in the first year, but not in the second year. It is possible that significant plant residues left from all three crops cumulatively from the previous year may have masked any RRPP effect on soil's BD and the C/N ratio. Also, aggregate stability may have been affected by SOC. As reported in a thorough literature review by Abiven et al. (2009) the effects of organic inputs over time on soil aggregate stability pose very complex relationships and no clear trend was evident based on rate of soil organic inputs or soil characteristics. This is an area of recommended additional research during the next phase of the study.

Yield results for all three crop species evaluated at each location suggested that the higher (4% v/v) application rate of RRPP produced significantly higher yields and can be recommended to farmers as means of improving their yields. Higher rates than the ones used in this study deserve further evaluation in subsequent studies, since the heavy metal content of this particular product seems not to cause environmental problems, based on soil samples analyses gathered during the first year of the study. Battaglia et al. (2003) reported interaction among paper pulp sludge and soil which resulted in the formation of new sorbing sites. As a result, Pb and Cd retention by the soil was significantly increased, thus reducing potential leaching.

The strong relationships found for leaf chlorophyll levels, as indicated by the high correlations between fresh weight and SPAD units, and between SPAD units and RRPP application rates, present valuable and useful information for improving management practices. Therefore, the SPAD meter appears to be a simple and reliable tool for optimizing management practices and can assist in further evaluation of RRPP effect on crop yields.

Results of VWC and ECa were not consistent. The results were most likely affected by collecting data under different soil moisture conditions with the WET instrument. Additional

thought is needed to determine how this simple and easy to use tool can be better used for management.

Amending soils with RRPP recycles carbon to the soil and converts a waste into valuable recourse. Increasing the soil carbon pool may have a beneficial effect on soil aggregate stability, may increase soil porosity and therefore improve infiltration and soil water holding capacity. Because of low mean annual precipitation and extended periods without any precipitation at all, in Greece most crops, and especially vegetables, require supplemental irrigation. In addition, irrigation water is becoming a limited commodity. Consequently, amending soils with RRPP may also reduce the amount of irrigation applied. Other studies show that, pulp application may also benefit the soil ecosystem (Beyer et. al., 1997) by increasing soil microbial activity and enhancing soil species spectrum and numbers in acidic soil restoration projects (Pierce and Boone, 1998). This agrees with Rotenberg et al. (2009) who reported that application of paper mill residual materials suppressed soil borne disease due to significant alteration in soil rhizobacterial communities and reduced counts in soil pathogens. It seems that the amount and quality of the organic matter in the soil influences the biotic and abiotic components in the soil, thus organic matter amendments may mediate soil functions and may contribute to crop health. As a result, less insecticide and fungicide may be needed to grow healthy crops, which in turn alleviate the pressure of those chemicals on the environment.

# **Recommendations**

Based on the positive results obtained from the first two years of this studyadditional research is planned to further evaluate RRPP as a soil amendment. During the first two years if the study we used LISA as much as possible. For the next two years we plan to evaluate the effect of RRPP under more conventional cultural practices in terms of pest control, irrigation and

fertilization. This may include but is not limited to: 1) using complete fertilizers for each crop, 2) varying irrigation rates and measuring the water use efficiency (WUE) 3) applying herbicides to control weeds and applying other pesticides as needed. Results will be compared to the LISA management scheme applied during the first two years. A comprehensive database will be established which will allow the analysis on a more extended time scale. This database will also be used to evaluate long-term effects on soil properties, as suggested by Aitken et al., 1998 and Douglas et al., 2003.

Soil sampling will include additional depths (down to 0.3-0.6 m) to investigate any potential effect of this material to the sub-soil, especially if deep plowing or other similar cultivation practices are used. Soil sample analyses from 0.3-0.6 m may reveal any nutrient and heavy metal leaching. Soil aggregate stability studies should be considered along with soil compaction readings to gain a more complete picture of the RRPP effect on soil physical properties. Higher application rates (8% and 12%) may be included in the evaluation, as well as application of the material in rough form (only partially ground), to reduce the overall cost of this material.

Other plant species such as legumes (i.e. soybeans) may be considered for introduction into the crop rotation cycle and evaluation. Relationships between chlorophyll levels and yield will be more intensively evaluated and the developed models will be validated against previous data. If funding is available, plant tissue samples will be collected and analyzed. The resulting models could be valuable management tools for each soil type and different crop species.

Additional soil analyses should be performed on the stored soil samples from the two previous years and the future soil samples. These analyses may include CEC, organic matter fractions, and incubation studies to investigate the decomposition rate of RRPP as reported by

Chantigny et al., (2000). This will allow us to better understand the effect of various RRPP application rates.

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### **APPENDICES**

### APPENDIX A

## 

P<sub>2</sub>O<sub>5</sub> analyses for AFS and Kolhiko (Analyses were provided from the Department of Agrochemistry and Plant Physiology, Faculty of Agriculture, University of Belgrade, Serbia, 2008 data)

Paper pulp residues have small to medium nutritive value. Ameliorating clay loam at AFS experimental area with 0, 1, 2, and 4% v/v pulp, did not affect significantly  $P_2O_5$  concentration at the end of the first growing season (Table A.1). First year's results indicate that the material cannot substitute for organic and/or inorganic  $P_2O_5$  sources. One way ANOVA by blocking replications showed that plots treated with 1% v/v RRPP had the largest  $P_2O_5$  concentration (3667.5 mg kg<sup>-1</sup>), followed by 2, 0, and 4% v/v application rates respectively.

Simple linear regression for first year's  $P_2O_5$  concentration revealed weak association with pulp application rates at the AFS (clay loam) site (Table A.2). Analysis of variance for simple linear regression (Table A.3) indicate that model's predictability on  $P_2O_5$  concentration is insignificant (p-value=0.975).

Conditioning sandy loam in 2008 (Kolhiko site), did not change soil P<sub>2</sub>O<sub>5</sub> mg kg<sup>-1</sup> significantly (Table A.1). P<sub>2</sub>O<sub>5</sub> concentrations at Kolhiko site are three folds lower compared to the averages observed at the AFS site. Elevated phosphorus concentrations at the AFS experimental site may have resulted from past animal manure spreading on the field. Commonly, animal wastes from industrial-style farms are high in phosphorus levels (Brady and Weil, 2008).

Spreading these manures on agricultural land may result in excessive soil phosphorus concentrations. Furthermore, clay and organic matter content are larger at clay loam compared to sandy loam. Those soil fractions are relatively rich in phosphorus. In alkaline soils highly soluble monocalcium phosphate will react with calcium carbonate to form di- and tri-calcium phosphates, which are highly insoluble and therefore not available to plant uptake. With time these phosphate forms can undergo further reactions and give various forms of apatites, whose solubility is further reduced.

RRPP contained calcium carbonate which may contribute further to phosphate immobilization in both sites (Table B.2). This may explain the lowest  $P_2O_5$  concentrations observed for 4% v/v application rates at AFS and Kolhiko site, as well. Measured pH at both sites gives values from neutral to moderately alkaline (up to 7.9), which supports the argument (Tables 4.12 and A.16).

Simple linear regression with replications as blocks (data from first year) indicates a small coefficient of determination ( $R^2$ =0.372). Therefore 37.2% of  $P_2O5_5$  variability at sandy loam experimental site is related to pulp application rates (Table A.4). Overall predictability of the model is insignificant as shown in Table A.5 (p-value=0.236).

K<sub>2</sub>O analyses for AFS and Kolhiko (Analyses were provided from the Department of Agrochemistry and Plant Physiology, Faculty of Agriculture, University of Belgrade, Serbia, 2008 data)

RRPP application (1, 2, and 4% v/v) at AFS site for the first year resulted in insignificant decrease in  $K_2O$  concentration (Table A.6). The largest arithmetical decrease was observed for the 4% v/v pulp rate (75 mg kg<sup>-1</sup>) compared to control. Plots treated with 2 and 1% v/v pulp had a statistically insignificant decrease of  $K_2O$  concentration, as well (12 and 37 mg kg<sup>-1</sup>,

respectively). We assume that potassium fixation was higher at the plots treated with RRPP because the material added additional CaCO<sub>3</sub> to the soil (Table 92), and raised pH (pH analysis from the University of Belgrade, Serbia indicated significant increase in treated plots). Lime addition into soils may increase potassium fixation by raising soil pH (Brady and Weil, 2008). As a result, hydroxyl aluminum ions and H<sup>+</sup> are neutralized or removed from the colloidal surfaces. Then, potassium monovalent cations are associated more tightly to colloidal surfaces, and therefore, potassium fixation potential increases significantly.

Simple linear regression for potassium concentrations (by blocking replications), gave low  $R^2$ =0.379 (Table A.7). Overall model predictability is insignificant since p-value=0.224 (Table A.8).

Data collected on the sandy loam (2008) were analyzed with one-way ANOVA and replications were blocked (Table A.6). The same pattern (as at AFS site) in K<sub>2</sub>O concentrations were observed at the Kolhiko experimental site. The lowest K<sub>2</sub>O concentrations (189.75 mg kg<sup>-1</sup>) were found in plots ameliorated with 4% v/v pulp. Amending soil with 2 and 1% v/v RRPP resulted in 259.25 and 254 mg kg<sup>-1</sup>, which were still lower from control (279.25 mg kg<sup>-1</sup>). Although, clay fraction is smaller in sandy loam compared to clay loam, pulp's liming properties raised pH (pH tables from Kolhiko) and promote probably potassium fixation. Still, K<sub>2</sub>O concentrations analysis did not showed statistically significant differences for plots received different pulp rates.

Simple linear regression indicates moderate  $R^2$  (0.452), and the overall p-value is large (p-value 0.128; Table A.9). Therefore, model's predictive value for  $K_2O$  concentration at the ameliorated sandy loam is insignificant (Table.10).

CaCO<sub>3</sub> analyses for AFS and Kolhiko (Analyses were provided from the Department of Agrochemistry and Plant Physiology, Faculty of Agriculture, University of Belgrade, Serbia, 2008 data)

Conditioning clay loam at the beginning of the first growing season with RRPP did not change  $CaCO_3$  concentration in the soil significantly (Table A.11).  $CaCO_3$  means comparison (Student's t-test,  $\alpha$ =0.05) for different application rates, showed that plots treated with the largest application rate (4% v/v) had the highest mean lime concentration, though it was not significant at  $\alpha$ =0.05.

Simple linear regression analysis (by blocking replications) for clay loam indicates very weak linear relationship ( $R^2$ =0.126) among lime concentration and the specific pulp application rates (0, 1, 2, and 4% respectively) (TableA.12). Similarly, large p-value=0.806 denotes insignificant predictability of the model on soil lime changes related to pulp conditioning (Table A.13).

One-way ANOVA analysis of data on  $CaCO_3$  concentration from Kolhiko experimental site (2008) showed increase in soil's  $CaCO_3$  directly proportional to pulp application rates (Table A.11), though not statistically significant at level of significance  $\alpha$ =0.05. This result was expected because of RRPP lime content and the low sandy loam's lime concentration (lime content at sandy loam was found 5 times lower compared to calcareous AFS soil).

After first application, RRPP rates are moderately associated (R<sup>2</sup>=0.412) with lime concentration in the sandy loam (Table A.14). Furthermore, analysis of variance for simple linear regression has large p-value=0.216, and therefore changes in lime concentration because of pulp application are not predictable with the simple regression model (Table A.15).

pH analyses for AFS and Kolhiko sites, (Analyses were provided from the Department of Agrochemistry and Plant Physiology, Faculty of Agriculture, University of Belgrade, Serbia, 2008 data)

For the first experimental year, soil reaction (pH) analyses were done for both experimental sites (AFS and Kolhiko) at the University of Belgrade, Serbia, as well. One-way ANOVA (by blocking replications) for pH analysis for extraction with water indicates statistically significant increase for 4% v/v pulp application rate compared to 0, 1, and 2% v/v (Table A.16).

Summary of fit for pH analysis (extraction with water) (Table A.17) revealed large  $R^2$ , and therefore 74.5% of the variability in soil pH is accounted for RRPP application rates. Furthermore, the overall linear model is statistically significant as indicated from the small p-value (Table A.18), though parameter estimates showed that treatments rates significantly affect soil pH when  $\alpha$ =0.08 (Table A.19). According to the model a 0.015 mol H<sup>+</sup> L<sup>-1</sup> increase in pH will follow a 1% increase in RRPP application rate.

Analysis of variance for pH extracted with KCl solution indicates insignificant change in soil pH accountable for pulp application rates at the AFS site (Table A.16). Simple linear regression for pH measured with KCl solution vs. pulp rates reveals small R<sup>2</sup> (TableA.20), and large p-value (table A.21). Therefore, for the AFS site (clay loam) overall model predictivity for pH changes related to pulp application rates is insignificant.

For 2008 pH data from Kolhiko (water extraction), one-way ANOVA indicates a statistically significant pH raise for all plots treated with pulp (Table A.16). This result was expected, since the pH buffering capacity of sandy loam is smaller than the one at clay loam. As a result, RRPP is a potential liming agent for acidic sandy loam soils. If applying the specific RRPP at alkaline sandy loams, then, monitoring soil pH is essential to keep pH at optimum levels.

Simple linear regression with replications used as blocks gave  $R^2$ =0.612 (Table A.22), and therefore 61.2% of pH variability is related to RRPP application rates. Overall p-value is significant at  $\alpha$ =0.05 (Table A.23), and we should expect a 0.06 raise in sandy loam pH for every 1% v/v increase in pulp application rate (Table A.24).

For 2008 pH data from Kolhiko, which were extracted with KCl solution, one-way ANOVA indicates a statistically significant pH raise for all plots treated with pulp (A.16). The results (arithmetical values) are close to AFS lab soil pH measurements for data collected in fall 2008, despite the differences in soil extraction methodologies. As a result, RRPP is a potential liming agent for acidic sandy loam soils, though successive RRPP application need close soil pH monitoring.

Simple linear regression analysis for pH measurements extracted with KCl resulted in small coefficient of determination (Table A.25), and the simple linear model has insignificant predictive value (Table A.26).

Heavy metals analysis (Analyses were provided from the Department of Agrochemistry and Plant Physiology, Faculty of Agriculture, University of Belgrade, Serbia, 2008 data)

For the first year, RRPP application to the clay loam did not change significantly concentration of heavy metals and other minerals that were tested (Tables A.27 to A. 30). This result was expected since the material is low in heave metals content (Table B.3). Also, Battaglia et al. (2007) reported in a study of an acidic soil polluted by heavy metals, mainly lead and zinc, that the addition of paper mill sludge induced a decrease in the mobile forms of these two metals. They hypothesized that presence of organic matter and some kaolinite (kalolinite is used as a filler during paper making, and is present in paper pulp residues) in the pulp sludge, were able to

bind the two metals very strongly. Furthermore, applying paper pulp sludge on an acidic soil polluted with heavy metals reduced the available metal forms (Calace et al., 2005).

Preliminary results in AFS soil for 2008 showed a decrease in Fe concentration (Table A.28), which was directly proportional to pulp application rates. The author assumes that the availability of the element to the plants increased as a result of pulp application. The assumption is supported by the work of Calace et al. (2006), which reported that application of paper mill sludge increased the availability of iron to *Hordeum distichum* plants in soils with low organic matter and alkaline pH (7.5 to 8.6).

Both soil concentrations in Pb, Ni, Cr, and Cu are higher compared to median from 3045 surface soils reported by Holmayer et al, 1993 (Brady, C. N., and R.R. Weil. 2008). At Kolhiko Cr mean concentration for control and 2% v/v treated plots (36.4 and 36.3 mg kg<sup>-1</sup> respectively) were lower from the median Cr concentration Holmayer et al. reported (37 mg kg<sup>-1</sup>). Plots where 1 and 4% v.v. pulp was applied showed Cr concentration slightly higher than 37 mg kg<sup>-1</sup> (Table A.27). Nevertheless, concentrations of all heavy metals tested in both sites were well below the maximum allowable cumulative pollutant loading for all treatments.

Table A.1 Mean  $P_2O_5$  (mg kg<sup>-1</sup>) for RRPP application rates at the end of the 1<sup>st</sup> growing season for both locations. Soil sample analyses at the University of Belgrade, Serbia.

Treatment	P <sub>2</sub> O <sub>5</sub> (AFS 2008)	P <sub>2</sub> O <sub>5</sub> (Kolhiko 2008)
RRPP% (v/v)±	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>
4	3365.00a†	141.00a†
2	3625.00a	167.50a
1	3667.50a	166.00a
0 (Control)	3487.50a	148.75a

 $\pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m. RRPP=Recycled residual Paper Pulp; AFS=American Farm School.

Table A.2 Summary of fit for  $P_2O_5$  (mg kg<sup>-1</sup>) at AFS 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.039	0.310	636.209	3536.250	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table A.3 Analysis of variance for  $P_2O_5$  (mg kg<sup>-1</sup>) at AFS 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

Source	DF	SS	MS	F Ratio	p-value
Model	4	182590.000	45647.500	0.113	0.975
Error	11	4452385.00	404762.273		
C. Total	15	4634975.00			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square, AFS=American Farm School.

Table A.4 Summary of fit for  $P_2O_5$  (mg kg<sup>-1</sup>) at Kolhiko 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.372	0.143	28.601	155.813	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response.

TableA.5 Analysis of variance for P<sub>2</sub>O<sub>5</sub> (mg kg<sup>-1</sup>) at Kolhiko 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

Source	DF	SS	MS	F Ratio	p-value
Model	4	5320.332	1330.083	1.626	0.236
Error	11	8998.105	818.010		
C. Total	15	14318.438			

Notes: The model was run as Simple Regression with replications used as blocks. SS=Sum of Squares; MS= Mean Square.

Table A.6 Mean K<sub>2</sub>O (mg kg<sup>-1</sup>) for RRPP application rates at the end of the 1<sup>st</sup> growing season for both locations. Analyses at the University of Belgrade, Serbia.

Treatment	K <sub>2</sub> O (AFS 2008)	K <sub>2</sub> O (Kolhiko 2008)
RRPP% (v/v)±	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>
4	952.50a†	189.750a†
2	1015.00a	259.250a
1	990.00a	254.000a
0 (Control)	1027.50a	279.250a

Notes: †Values followed by the same letter are not statistically significant at p-value<0.05 (Student's t-test).

 $\pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m. RRPP=Recycled residual Paper Pulp; AFS=American Farm School.

Table A.7 Summary of fit for  $K_2O$  (mg kg<sup>-1</sup>) at AFS 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

$\mathbb{R}^2$	R <sup>2</sup> Adj	RMSE	MR	Observations
0.379	0.154	62.743	996.250	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table A.8 Analysis of variance for  $K_2O$  (mg kg<sup>-1</sup>) at AFS 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

Source	DF	SS	MS	F Ratio	p-value
Model	4	26471.429	6617.857	1.681	0.224
Error	11	43303.571	3936.688	0.224	
C. Total	15	69775.000			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table A.9 Summary of fit for  $K_2O$  (mg kg<sup>-1</sup>) at Kolhiko 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.452	0.252	80.014	245.563	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response.

Table A.10 Analysis of variance for K<sub>2</sub>O (mg kg<sup>-1</sup>) at Kolhiko 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

Source	DF	SS	MS	F Ratio	p-value
Model	4	58047.404	14511.851	2.267	0.128
Error	11	70424.534	6402.230	0.128	
C. Total	15	128471.938			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square.

Table A.11 Mean CaCO<sub>3</sub> (mg kg<sup>-1</sup>) for RRPP application rates at the end of the 1<sup>st</sup> growing season for both locations. Analyses at the University of Belgrade, Serbia.

Treatment	CaCO <sub>3</sub> (AFS 2008)	CaCO <sub>3</sub> (Kolhiko 2008)
RRPP% (v/v)±	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>
4	8610.00a†	525.00a†
2	6930.00a	455.00a
1	6650.00a	350.00a
0 (Control)	7035.00a	140.00a

Notes: †Values followed by the same letter are not statistically significant at p-value<0.05 (Student's t-test).

 $\pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m.

RRPP=Recycled residual Paper Pulp; AFS=American Farm School.

Table A.12 Summary of fit for CaCO<sub>3</sub> (mg kg<sup>-1</sup>) at AFS 2008 vs treatments. Analyses at the University of Belgrade, Serbia.

$\mathbb{R}^2$	$R^2Adj$	RMSE	MR	Observations
0.126	-0.191	3221.065	7306.250	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table A.13 Analysis of variance for  $CaCO_3$  (mg kg<sup>-1</sup>) at AFS 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

Source	DF	SS	MS	F Ratio	p-value
Model	4	16495150.0	4123787.50	0.397	0.806
Error	11	114127825	10375256.8		
C. Total	15	130622975			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table A.14 Summary of fit for CaCO<sub>3</sub> (mg kg<sup>-1</sup>) at Kolhiko 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

$\mathbb{R}^2$	R <sup>2</sup> Adj	RMSE	MR	Observations
0.412	0.176	277.822	382.667	15

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response.

Table A.15 Analysis of variance for CaCO<sub>3</sub> (mg kg<sup>-1</sup>) at Kolhiko 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

Source	DF	SS	MS	F Ratio	p-value
Model	4	540044.277	135011.069	1.749	0.216
Error	10	771849.057	77184.906		
C. Total	14	1311893.33			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square.

Table A.16 Mean pH (extraction with H<sub>2</sub>O and KCl) for RRPP application rates at the end of the 1<sup>st</sup> growing season for both sites. Soil sample analyses at the University of Belgrade, Serbia.

Treatment	pH pH (AFS 2008) (Kolhiko 2008)		pH (AFS 2008)	pH (Kolhiko 2008)
	H	[2O	K	Cl
-RRPP% (v/v)±-	mol H <sup>+</sup> L <sup>-1</sup>	mol H <sup>+</sup> L <sup>-</sup>	$$ mol $H^+L^-$	mol H <sup>+</sup> L <sup>-</sup>
4	7.928a†	7.853a†	7.338a†	7.143a†
2	7.853b	7.775a	7.265a	7.183a
1	7.883ab	7.845a	7.320a	7.183a
0 (Control)	7.860ab	7.535b	7.255a	6.652b

RRPP=Recycled residual Paper Pulp; AFS=American Farm School.

Table A.17 Summary of fit for pH (Analysis with H<sub>2</sub>O) at AFS 2008 vs treatments. Analyses at the University of Belgrade, Serbia.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.745	0.652	0.046	7.881	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table A.18 Analysis of variance for pH (Analysis with H<sub>2</sub>O) at AFS 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.068	0.017	8.014	0.003*
Error	11	0.023	0.002		
C. Total	15	0.091			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Values with (\*) are statistically significant.

 $<sup>\</sup>pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m.

Table A.19 Parameter estimates for pH (Analysis with H<sub>2</sub>O) at AFS 2008 vs. treatments. (probably not needed). Analyses at the University of Belgrade, Serbia.

Term	Estimate	SE	t Ratio	Prob> t
Intercept	7.854	0.018	440.871	0.000*
Treatments	0.015	0.008	1.957	0.076
Rep-Block[1]	-0.001	0.020	-0.031	0.976
Rep-Block[2]	-0.083	0.020	-4.173	0.002*
Rep-Block[3]	-0.006	0.020	-0.282	0.783

Note: Values indicated with (\*) are statistically significant at level of significance  $\alpha$ =0.05.

The model was run as Simple Regression with replications used as blocks.

SE=Standard Error; AFS=American Farm School.

Values indicated with (\*) are statistically significant.

Table A.20 Summary of fit for pH (Analysis with KCl) at AFS 2008 vs treatments. Analyses at the University of Belgrade, Serbia.

$\mathbb{R}^2$	$\mathbb{R}^2 \mathbf{Adj}$	RMSE	MR	Observations
0.270	0.005	0.079	7.294	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response; AFS=American Farm School.

Table A.21 Analysis of variance for pH (Analysis with KCl) at AFS 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.025	0.006	1.018	0.440
Error	11	0.069	0.006		
C. Total	15	0.094			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square; AFS=American Farm School.

Table A.22 Summary of fit for pH (Analysis with H<sub>2</sub>O) at Kolhiko 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

$\mathbb{R}^2$	$R^2 Adj$	RMSE	MR	Observations
0.612	0.472	0.159	7.752	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response.

Table A.23 Analysis of variance for pH (Analysis with H<sub>2</sub>O) at Kolhiko 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.437	0.109	4.347	0.024*
Error	11	0.277	0.025		
C. Total	15	0.714			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square.

Values with (\*) are statistically significant (p<0.05).

Table A.24 Parameter estimates for pH (Analysis with H<sub>2</sub>O) at Kolhiko 2008 vs. treatments. (probably not needed). Analyses at the University of Belgrade, Serbia.

<u> </u>	.,,			
Term	Estimate	SE	t Ratio	Prob> t
Intercept	7.643	0.061	124.469	0.000*
Treatments	0.062	0.027	2.311	0.041*
Rep-Block[1]	0.228	0.069	3.323	0.007*
Rep-Block[2]	-0.032	0.069	-0.464	0.652
Rep-Block[3]	-0.139	0.069	-2.030	0.067

Note: Values indicated with (\*) are statistically significant at level of significance  $\alpha$ =0.05.

The model was run as Simple Regression with replications used as blocks.

SE=Standard Error.

Table A.25 Summary of fit for pH (Analysis with KCl) at Kolhiko 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

$\mathbb{R}^2$	$R^2Adj$	RMSE	MR	Observations
0.406	0.190	0.274	7.040	16

Notes: The model was run as Simple Regression with replications used as blocks.

RMSE=Root Mean Square Error; MR= Mean of Response.

Table A.26. Analysis of variance for pH (Analysis with KCl) at Kolhiko 2008 vs. treatments. Analyses at the University of Belgrade, Serbia.

Source	DF	SS	MS	F Ratio	p-value
Model	4	0.564	0.141	1.878	0.185
Error	11	0.826	0.075		
C. Total	15	1.390			

Notes: The model was run as Simple Regression with replications used as blocks.

SS=Sum of Squares; MS= Mean Square.

Table A.27 Mean Cr and Co concentration (mg kg<sup>-1)</sup> for RRPP application rates at the end of the 1<sup>st</sup> growing season for both locations. Soil sample analyses at the University of Belgrade, Serbia.

Treatment	Cr (AFS 2008)	Cr (Kolhiko 2008)	Co (AFS 2008)	Co (Kolhiko 2008)
RRPP% (v/v)±	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>
4	49.675a†	38.825a†	11.370a†	9.325a†
2	53.125a	36.325a	11.450a	8.695a
1	53.790a	37.150a	11.595a	8.983a
0 (Control)	54.615a	36.435a	11.795a	9.160a

Table A.28 Mean Cu and Fe concentration (mg kg<sup>-1</sup>) for RRPP application rates at the end of the 1<sup>st</sup> growing season for both locations. Soil sample analyses at the University of Belgrade, Serbia.

Tweetment	Cu	Cu	Fe	Fe
Treatment	(AFS 2008)	(Kolhiko 2008)	(AFS 2008)	(Kolhiko 2008)
RRPP% (v/v)±	m	g kg <sup>-1</sup>		%
4	26.040a†	24.288a†	2.125a†	2.560a†
2	26.328a	21.850a	2.170a	2.483a
1	26.385a	23.350a	2.288a	2.665a
0 (Control)	26.230a	22.350a	2.343a	2.498a

Notes: †Values followed by the same letter are not statistically significant at p-value<0.05 (Student's t-test).

Table A.29 Mean Mn and Ni concentration (mg kg<sup>-1</sup>) for RRPP application rates at the end of the 1<sup>st</sup> growing season for both locations. Soil sample analyses at the University of Belgrade, Serbia.

Treatment	Mn (AFS 2008)	Mn (Kolhiko 2008)	Ni (AFS 2008)	Ni (Kolhiko 2008)
RRPP% (v/v)±	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>
4	709.250a†	365.250a†	41.935a†	21.370a†
2	735.000a	339.000a	42.613a	19.745a
1	737.250a	349.250a	42.530a	20.493a
0 (Control)	732.500a	346.250a	43.023a	20.468a

Notes: †Values followed by the same letter are not statistically significant at p-value<0.05 (Student's t-test).

 $<sup>\</sup>pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m. RRPP=Recycled residual Paper Pulp; AFS=American Farm School.

 $<sup>\</sup>pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m. RRPP=Recycled residual Paper Pulp; AFS=American Farm School.

<sup>± (</sup>v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m. RRPP=Recycled residual Paper Pulp; AFS=American Farm School.

Table A. 30 Mean Pb and Zn concentration (mg kg<sup>-1</sup>) for RRPP application rates at the end of the 1<sup>st</sup> growing season for both locations. Soil sample analyses at the University of Belgrade, Serbia.

Treatment	Pb	Pb	Zn	Zn
Treatment	(AFS 2008)	(Kolhiko 2008)	(AFS 2008)	(Kolhiko 2008)
RRPP% (v/v)±	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>
4	24.238a†	11.650a†	36.090a†	33.055a†
2	23.463a	11.475a	37.835a	32.835a
1	23.175a	11.975a	37.550a	33.910a
0 (Control)	22.950a	11.988a	37.468a	33.660a

 $<sup>\</sup>pm$  (v/v) Volume of material applied to volume of soil calculated to a depth of 0.15m. RRPP=Recycled residual Paper Pulp; AFS=American Farm School.

### APPENDIX B

# RECYCLED RESIDUAL PAPER PULP PROPERTIES (RRPP)-PHYSICAL AND CHEMICAL

### Bulk density determination of ground air dried recycled residual paper pulp

RRPP that was applied to the soils was air dried for 15 days to minimize the moisture content and ground into fine particles 2-4 mm at the CaO Hellas S.A plant. The grounded pulp was packed into 500 kg sacks, and was shipped to the AFS site.

For measurements of BD for the RRPP, four stainless steel cylinders of 100 cm<sup>3</sup> volume (sub-replications) were assigned into 4 replication groups, totaling 16 samples (replications). Randomly selected sacks were sampled, and the samples were moved into the laboratory for BD measurement. The stainless cylinders lightly packed to the volume of the ring with the ground pulp oven dried for 24 hrs at 105 °C. The following day, each cylinder with the oven dry pulp was re-weighed to determine ground pulp's water content. Subtracting water mass and cylinder mass from the initial mass (cylinder and ground air dried pulp mass) gave oven dry mass. BD of the ground pulp was calculated as follows:

BD=mass (oven dried ground pulp)/V (stainless steel cylinders) in Mg m<sup>-3</sup>

One way ANOVA by blocking replications was carried, and the results are shown in table B.1. Means between replications did not shown statistically significant differences ( $\alpha$ =0.05) as was expected. The average BD for ground RRPP was equal to 0.637 Mg m<sup>-3</sup>.

### Chemical analyses of recycled residual paper pulp from different laboratories

Additional analysis of RRPP samples was carried out at different laboratories. This section includes comparison tables of those analyses, and established concentration limits in EU and the United States for heavy metals in sludge applied to agricultural lands. RRPP sludge analyses for heavy metal from all laboratories and different sampling times showed that inorganic pollutants are well below the established thresholds for sludges applied to agricultural lands. Directive 86/278/EEC (EC, 1986) regulates heavy metal in sludges in EU, while, USEPA has set the limits for the United States.

Table B.1 Mean BD for RRPP material used to ameliorates experimental plots in both years (2008 and 2009).

Means of sub- replications within Replication	BD
	Mg m <sup>-3</sup>
D	0.653a†
A	0.643a
В	0.628a
C	0.625a
	Grand Average
	0.637

One-way ANOVA by blocking replications was carried out. In each replication we had 4 samples of RRPP.

RRPP=Recycled residual Paper Pulp; BD= Bulk Density; A, B, C, and D=replications contained four sub-replications.

Table B.2 Chemical analysis of RRPP from various labs.

Parameter	CIP Nov 2007	AGROLAB Jul 13 <sup>th</sup> 2007	AGROLAB Jul 19 <sup>th</sup> 2007	AGROLAB Aug 7 <sup>th</sup> 2007	AGROLAB Averages	SPWL Nov 2008
pH (1:5 <sup>w</sup> / <sub>y</sub> )	N/A	7.9	7.8	8.0	7.90	
EC (dS m <sup>-1</sup> )	N/A	0.577	0.633	0.934	0.70	
CaCO <sub>3</sub> (% d.m.)	N/A	47.52	4.2	36.4	29.37	
SOM (% d.m.)	10.6	32.5	22	37.3	30.60	
Total C(% d.m.)	6.17	21.9	11	18.6	17.17	18.02
C/N	N/A	44.1	26.8	80.9	50.60	60.06
$N-NO_3 (mg kg^{-1})$	N/A	284	112	426	274.00	
$N-NH_4$ (mg kg <sup>-1</sup> )	N/A	3150	2497	649	2098.67	
Total N(mg kg <sup>-1</sup> )	N/A	4969	3986	1680	3545.00	3000
$PO_4^{-3} (mg \ kg^{-1})$	N/A	570	398	573	513.67	
Ca (mg kg <sup>-1</sup> )	N/A	185913	254000	194650	211521.00	179600
$Mg (mg kg^{-1})$	N/A	2703	3675	2272	2883.33	3015
$Mn (mg kg^{-1})$	N/A	N/A	N/A	N/A	N/A	90.15
$K (mg kg^{-1})$	N/A	558	155	149	287.33	849.8
Na (mg kg <sup>-1</sup> )	N/A	1153	635	465	751.00	466.8
$S (mg kg^{-1})$	N/A	N/A	N/A	N/A	N/A	600.7

Notes: d.m.=dry mater; N/A=Not Available. CIP is a soil analytical laboratory in Germany; Agrolab is a soil analytical Laboratory in Thessaloniki, Greece; .SPWL=Soil, Plant, and Water Laboratory, in Athens, GA, USA.

Table B.3 Heavy metal RRPP content from various labs and the regulatory limits in sewage sludge applied to farmlands.

Parameter	CIP Nov 2007	AGROLAB Av. Jul-Aug 2007	SPWL Nov 2008	Max Sludge Concentrations USEPA	CT Sludge Concentrations European Directive 86/278
	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>	mg kg <sup>-1</sup>
Al	11890	N/A	N/A	N/A	N/A
Fe	N/A	379.33	2537	N/A	N/A
Cu	63.4	65.03	66.61	N/A	1750
Mn	N/A	46.97	N/A	N/A	N/A
Zn	52.3	204.47	66.83	7500	2500-4000
В	N/A	22.67	3.19	N/A	N/A
Pb	< 0.1	1.60	< 2.5	840	750-1200
Cd	< 0.5	0.56	2.01	85	20-40
Ni	< 0.1	2.30	4.17	420	300-400
Co	N/A	0.82	N/A	N/A	N/A
Cr	13.4	9.29	13.79	3000	N/A
As	4.6	0.63	N/A	75	N/A
Hg	< 0.1	< 0.50	N/A	57	16-25
Ag	0.58	N/A	N/A	N/A	N/A
Si	N/A	N/A	358.2	N/A	N/A

Notes: d.m.=dry mater; N/A=Not Available, Max=Maximum, CT=Critical Threshold. CIP is a soil analytical laboratory in Germany; Agrolab is a soil analytical Laboratory in Thessaloniki, Greece; .SPWL=Soil, Plant, and Water Laboratory, in Athens, GA, USA.

### **APPENDIX C**

### **WEATHER DATA**

Table C.1 Historical average temperatures and precipitation data for Thermi, Thessaloniki, Greece (1931-1960).

Climatic data	Jan	Feb	Mar	April	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Annual
Max. Temp.†	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
Min. Temp.	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	N/A	
Mean Temp.	5.5	7.1	9.6	14.5	19.6	24.7	27.3	26.8	22.5	17.1	12	7.5	$16.1 \pm$
Precipitation¥	44	34	35	36	40	33	20	14	28	55	56	44	$449\P$

Notes: †Temperatures in °C; ¥ precipitation in mm; ± Annul mean temperature; ¶ Yearly cumulative precipitation; N/A=Not available.

Table C.2 Historical average temperatures and precipitation data for Thermi, Thessaloniki, Greece (1961-1990).

Climatic data	Jan	Feb	Mar	April	May	Jun	Jul	Aug	Sep	Oct	Nov	Dec	Annual
Max. Temp.†	9	11	14	19	25	29	31	31	27	21	15	11	
Min. Temp.	1	2	4	7	12	16	18	18	15	11	7	3	
Mean Temp.	5	7	10	14	20	24	27	27	22	16	11	7	$15.75\pm$
Precipitation¥	37	40	46	36	44	32	26	26	26	41	58	53	$460\P$

Notes: †Temperatures in °C; ¥ precipitation in mm; ± Annul mean temperature; ¶ Yearly cumulative precipitation.

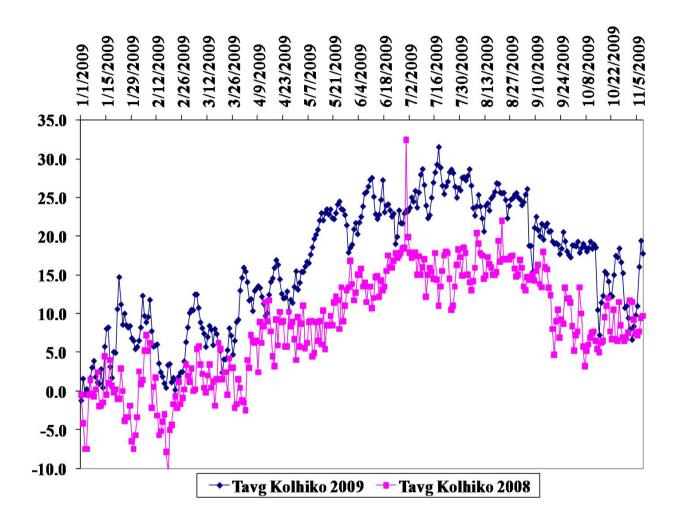


Figure C.1 Average daily temperatures (°C) for Kolhiko (2008 & 2009).

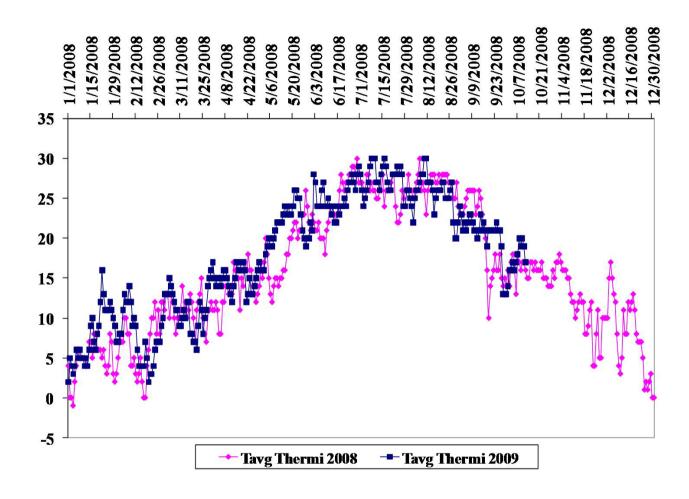


Figure C.2 Average daily temperatures (°C) for AFS (2008 & 2009).

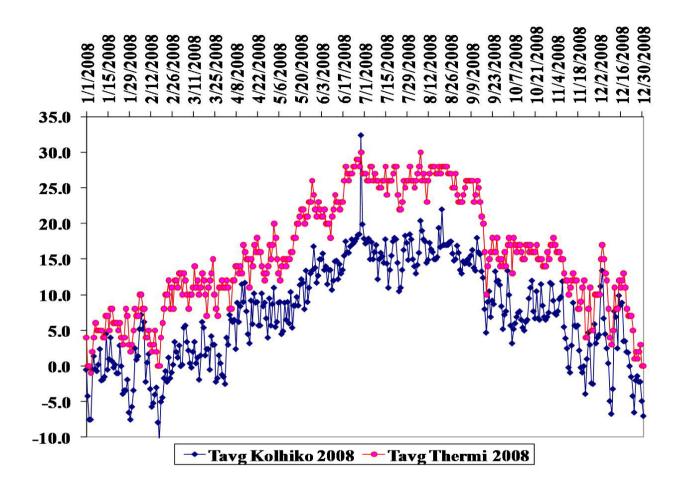


Figure C.3 Average daily temperatures at (°C) Kolhiko and AFS (2008).

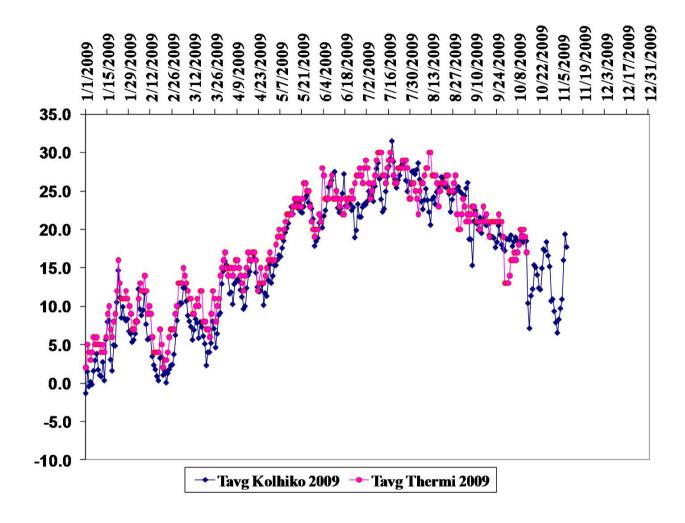


Figure C.4 Average daily temperatures at (°C) Kolhiko and AFS (2009).