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The possible replacement of calcite by calcareous sludge of paper mill in amending cultivated soil of white mulberry (Ichinoife variety)

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Abstract

Paper mill sludge is produced as a by-product of paper production such that disposal of this material presents a problem for the mill. Disposal by land filling, the most common disposal method, is costly and faces increasingly stringent environmental regulations. In this study, the possibility of using paper sludge lime instead of calcite lime had been investigated in berry orchard improvement under cultivation of sericulture in two phases. In the first phase, the effect of different amounts of paper mill sludge on some chemical properties of soil was investigated in an incubation study at the period of 90 days. The results showed that the proportional amount of paper sludge increased pH and decreased soil iron and manganese, but will lead to increased soil phosphorus. In a field experiment (second phase), control treatment and three treatments in the amounts of lime sludge applied at the rate of 2.5, 5 and 10 ton/ha; and a treatment with 2.5 tons/ha of calcite in the form of a completely randomized block design in the mulberry orchard of the Silkworm Research Center had been carried out. The results indicated that the amount of paper sludge, 2.5 and 5 tons/ha, along with calcite lime treatments, leaf dry matter yield and gross protein content in leaves significantly increased compared to control, but with regard to yield, percentage of protein and nutrient uptake, treatment of sludge lime were more appropriate than calcite lime treatment.

Keywords: Acid soil, Amendment, Environment, Lime, Silk worm.

Introduction

The most common management practice to ameliorate acid soils is the surface application of lime and other calcareous materials (Bolan *et al.,* 2003). The main aim of soil liming is to neutralize acidic inputs and for recovering the buffering capacity to the soil (Ulrich, 1983). Applications of industrial wastes as fertilizer and soil amendment have become popular in agriculture. One of these wastes is calcareous sludge of the paper industry. Paper mill sludge is produced as a by-product of paper production such that disposal of this material presents a problem for the mill (Battaglia *et al.,* 2007; Calace *et al.,* 2005; Mahmood and Elliot, 2006). Disposal by land filling, the most common disposal method, is costly and faces increasingly stringent environmental regulations (Feldkinchner *et al.,* 2003). Lime sludge is the solid waste produced as part of the process that turns wood chips into pulp for paper. The major component of lime mud is calcium carbonate ($CaCO_3$) and it is estimated that about 0.47 m^3 of lime mud is generated to produce 1ton of pulp (Wirojanagud et al., 2004). Mohammadi Torkashvand (2010) compared the effect of sulfuric acid and paper mill sludge on the produced compost quality of municipal wastes. He found that both amendments increased the agronomic value of the produced compost quality, but using sulfuric acid is suitable in fewer amounts (20 ml/kg fresh organic wastes; decomposable municipal wastes). The paper mill sludge had more effect on total nitrogen of the produced compost than the sulfuric acid. For land application of sludge produced from pulp mills, Simpson et al. (1982) reported that combined kraft paper mill secondary sludge-fly ash applied at a rate of 108 metric dry ton ha^{-1} significantly increased the yield of fescue and corn. A four years field study in Alberta (Macyk, 1996) recommended an agronomically sound decomposed pulp mill sludge application rate of 40–80 ton ha^{-1} for brome grass. Kannan and Oblisami (1990) also concluded in a similar research that paper alkaline waste along pulp with paper in irrigating sugarcane fields is leading to reduced growth. High consumption of sludge (10 ton/ha) also caused reduced plant yield. Leon *et al.* (2006) during a research concluded that the use of paper sludge as soil modifiers significantly caused decreased rot in beans in sludge treatments compared with control. They found that the decrease in disease resulted from a change in properties of soil biology (Leon *et al.,* 2006). Curnoe *et al.* (2006) identified the positive effects of factory lime sludge paper on maize yield. Gaskin and Morris (2004) indicated that lime mud has the potential to be used as an agricultural liming material because of its capability to neutralize soil acidity (increased soil pH) and to add calcium and magnesium to the soil. Although high moisture content of lime mud creates more shipping and handling difficulties than typical dry agricultural liming materials (Mahmoudkhani et al., 2004), this obstacle can be overcome as sludge dewatering technology improves (Chen et al., 2002 and Yin et al., 2004). The objectives of the present study were (i) to determine the effect of paper mill sludge on some chemical properties of soil (pH, EC, AB-DTPA extractable P, K, Fe, Mn and Zn) and growth of berries as compared with calcite lime and, (ii) to evaluate the value of the waste as an agricultural lime material.

Table 1. Some properties of soils in incubation and field phases

Soil properties	Incubation phase	Field phase
pH	6.2	6.3
ECe (dS/m)	1.1	0.32
Total N (%)	0.085	0.105
Available P (mg/kg)	15.6	17.2
Available K (mg/kg)	145.2	168.2
Organic Matter (%)	0.92	1.12
Texture	Clay Loam	Clay Loam

Table 2. The effect of treatments on some chemical properties of soil

Treatments	pH	EC (dS/m)	P	K	Fe	Zn	Mn
			mg/kg				
L_0	6.04 f	0.37 d	13.1 bc	126.4 a	202.3 a	1.8 c	17.3 a
$L_{0.1}$	6.19 f	0.58 c	11.5 a	125.8 a	191.0 ab	2.1 bc	15.1 ab
$L_{0.2}$	6.92 e	1.08 c	16.0 a	122.6 ab	182.1 b	2.6 ab	14.2 bc
$L_{0.5}$	7.29 d	1.31 c	16.7 a	116.5 abcd	164.7 c	3.0 a	12.6 cd
L_1	7.24 d	1.63 b	15.3 ab	108.5 d	142.5 d	2.6 ab	10.8 de
L_2	7.63 c	1.82 b	16.5 a	113.1 bcd	120.0 e	2.8 ab	9.5 e
L_4	8.25 b	1.96 b	16.0 a	111.3 cd	86.9 f	2.5 ab	9.4 e
L_8	8.74 a	2.76 a	14.3 ab	119.6 abc	87.8 f	2.4 abc	8.4 e

LSD (least significant difference) shows the significant difference ($\rho = 0.05$) among the different treatments.
Values followed by the same letters in each column are not significantly different at the 0.05 level (least significant difference

Results

The chemical composition of the paper mill sludge showed that this compound contained about 58.4% calcium carbonates equivalent and a pH about 13.2 (pH of 1:2.5 dry paper mill sludge/water suspension), and 4.12, 2.35, 7.54, 3.25 and 28.6 $mg.kg^{-1}$ of Zn, Cu, Cr, Cd and Pb, respectively.Table 2 shows the effect of added paper mill sludge on soil pH and EC; and the AB-DTPA extractable P, K, Fe, Mn and Zn. Paper mill sludge remarkably increased pH, which was proportional to the application rate of paper mill sludge. Increase in pH was 0.15 units for $L_{0.1}$, 0.88 units for $L_{0.2}$, 1.2 units for $L_{0.5}$, 1.25 units for L_1, 1.59 units for L_2, 2.21 units for L_4 and 2.7 units for L_8 compared to the control (L_0). Paper mill lime sludge increased soil electrical conductivity, 0.71, 0.93, 1.23, 1.45, 1.59 and 2.39 times compared with control treatments in $L_{0.2}$, $L_{0.5}$, L_1, L_2, L_4 and L_8 treatments, respectively. The application of paper mill sludge resulted in 0.2-8% increased in AB-DTPA extractable P, but this increase was not significant in 1% and 8% paper mill sludge. Paper mill sludge resulted in decreased AB-DTPA-extractable K in L_1, L_2 and L_4 treatments, remarkably. Paper mill sludge remarkably decreased available Fe, which was proportional to the application rate of paper mill sludge. Of course, this decrease in Fe of $L_{0.1}$ treatment, rather than in the control, sample was not significant. Available Zn increased by paper mill sludge, but this increase in $L_{0.1}$ and L_8 treatments was significant. Paper mill sludge has remarkably decreased AB-DTPA extractable Mn rather than in the control sample that this is considerable in further amounts of sludge. The effect of treatments on berry yield is shown in Table 3. Berry leaf dry matter yield increased significantly ($P \leq 0.05$) in 2.5 and 5 ton/ha of paper mill sludge treatments rather than in the control sample. Yield was 1.36 and 1.55 times higher than in the control (L_0). Table 4 shows the effect of treatments on protein and nutrient concentration in berry leaves in the field experiment. Paper lime sludge and calcite lime treatments increased leaf protein, significantly. Sericulture production depends on the amount of leaves proteins that sludge caused an increase in the protein and yield of leaves. Nitrogen concentration in leaves was greater in $L_{2.5}$ and L_c treatments than in the control plants. P-leaf concentration decreased in L_5, L_{10} and L_c treatments than in the control, significantly, but K-leaf concentration has remarkably increased in $L_{2.5}$, L_5 and L_c. The concentration of Mn in leaves decreased significantly in calcite treatment, but it increased in 2.5 ton/ha paper mill sludge treatment significantly. Paper mill sludge treatments caused an increase in Zn concentration in leaves more remarkably than in the control sample.The effect of treatments on nutrients uptake of berries in the field experiment is shown in Table 5. Nitrogen, phosphorus and potassium uptake of berries was greater in treatments containing 2.5 and 5 ton/ha of paper mill sludge than in the control plants. Mn uptake increased in $L_{2.5}$ and L_5 treatments as compared with control. Residual effects of treatments on AB-DTPA-extractable P, K, Fe, Mn and Zn of soil are shown in Table 6. Residual AB-DTPA-extractable P, K, Fe and Zn was greater in treated soil by 5 ton/ha of paper mill sludge than in the control after harvesting, but AB-DTPA-extractable Mn decreased in this treatment.

Discussion

Increase in soil pH by paper mill sludge indicates the usefulness of paper mill sludge as a liming agent for amelioration of acid soils, but the further amount of this by-product is not suitable as a soil amendment, particularly in primary stages (Mohammadi A. Torkashvand, 2010). Similar results were obtained by Hea *et al.* (2009) for the use of paper mill lime mud as a liming agent and its effect on soil pH and ryegrass growth. Soil electrical conductivity increased by the use of paper mill sludge, but this increase in EC did not change the salinity order of the soil, consequently, taking into consideration electrical conductivity, the application of paper mill sludge in lower amounts does not affect the growth of many crops.Soil AB-DTPA-extractable P increased in paper mill sludge treatments. High pH values associated with high quantities of Ca probably facilitate precipitation of P as calcium phosphates, thus, limiting the availability of P to the

Table 3. The effect of treatments on growth of berry in field experiment

Treatment	Leaf dry Matter (kg)	Shoots weight (kg)	Shoots + leaves weight (kg)	Longest shoot (m)	Leaf moisture (%)
L_0	3.97 d	4.16 a	9.11 ab	1.65 a	72.0 a
$L_{2.5}$	5.42 ab	4.35 a	9.73 ab	1.73 a	71.8 a
L_5	6.16 a	4.49 a	10.62 ab	1.81 a	73.3 a
L_{10}	4.26 dc	2.96 b	10.60 ab	1.41 a	72.2 a
L_C	4.96 bc	2.83 b	7.20 b	1.67 a	71.8 a

Table 4. The effect of treatments on protein and nutrients concentration of berry leaves

Treatment	Protein	N	P	K	Fe	Mn	Zn
	(%)				(mg/kg)		
L_0	1.29 c	0.21 c	0.44 a	0.45 c	50.0 ab	36.1 b	94.3 c
$L_{2.5}$	1.62 ab	0.26 ab	0.46 a	0.73 a	42.1 b	46.2 a	172.1 a
L_5	1.52 abc	0.24 abc	0.29 bc	0.60 b	45.5 ab	32.2 b	130.2 b
L_{10}	1.43 bc	0.23 bc	0.20 c	0.45 c	57.1 a	39.4 bc	136.8 b
L_C	1.75 a	0.28 a	0.30 b	0.57 b	35.4 b	20.7 c	61.0 c

plant. In contrast, a similar increase in pH due to lime application, as happened in the present study, may cause some solubilization of P from Fe–P and Al–P complexes, thus, increasing P availability as was suggested by McCants and Woltz (1967). It seems that soil P concentration increased due to the solubilization of P from Fe–P and Al–P complexes, but it did not change in incubation experiments which may be due to an interaction between precipitation and solubilization of P by liming. The decrease in soil potassium may be due to K fixation. Mohammadi Torkashvand and Sedaghathoor (2007) reported that the use of a calcareous by-product of the steel industry as a liming agent reduced K content in an acid soil. They stated that this decrease in soil K might be due to the potassium fixation. Extractable Fe decreased in proportion to the application rate of paper mill sludge. Extractable Fe concentration depends on initial pH of soil. Increase in soil pH in the range of 7.4-8.5 decreased Fe level. It was found that Fe was precipitated as $Fe(OH)_3$ due to the increased pH (Norvell and Lindsay, 1982). Similar results were found by Mohammadi Torkashvand and Sedaghathoor (2007) with the use of calcareous converter slag in acid soils. Increasing zinc concentration in soil can be due to the added Zn by paper mill sludge (the used sludge contains 4.12 $mg.kg^{-1}$). Decrease in Mn by paper mill sludge treatments can be due to increasing soil pH and a decrease in the solubilization of Mn compounds. Many studies have shown that the liming improved the growth of many crops cultivated on acid soils such as red clover (Steiner and Alderman, 2003), wheat and barley (Tang *et al.,* 2003) and peanut (Chang and Sung, 2004). The results show that correction of soil acidity leads to improved soil conditions for growth of berries and this was causing an increase of leaf dry matter yield. Hea *et al.* (2009) found that the ryegrass yield had a better response to the application of paper lime sludge in the amounts between 4.51 and 9.01 $ton.ha^{-1}$ of lime sludge. Dutta and Boissya (1997) in an experimental study concluded that increasing the amount of alkaline wastewater of a paper factory led to reduced growth of rice seedlings, but Pantazis *et al.* (2009) found that the application of paper alkaline waste with paper calcareous sludge increased the plant growth in a greenhouse experiment. Increase of leaf protein in sludge treatments occurs because of the increase of leaf N concentration in this treatment. Because leaf nitrogen concentration is more in calcite lime treatment than in sludge treatment, so the percentage of protein in the leaves in calcite lime treatments is even higher in sludge treatment, hence, according to the leaves' yields, in the treated soils with 2.5 and 5 ton sludge, is more important in comparison with calcite lime. Increasing the N uptake in treatments of paper sludge and lime calcite may be because of the increasing concentration of nitrogen in leaves dry matter and increase in the yield of plants under these treatments. Increase of Nitrogen concentration in plants may be because of immediate availability of this element in soil due to increases in soil microbial activity and nitrogen mineralization due to improving soil acidity conditions. Nunes *et al.* (2008) during a greenhouse study to evaluate the potential of paper lime sludge as a liming factor on the growth of wheat in two soils of Cambisols and Arenosol concluded that the use of it significantly increases the soil pH, total nitrogen, available phosphorus and exchangeable potassium. They introduced this by-product as a calcareous factor in amending acidic soils which are better for growth of grain crops along with magnesium fertilizers. The further uptake of phosphorus in 2.5 ton/ha of paper mill sludge treatment is due to the higher concentration of phosphorus in leaves and also higher plant yield (dry leaves). The lower uptake of phosphorus in other treatments of paper mill sludge and calcite lime treatment may be due to the lower concentration of phosphorus in leaves. It is likely that the increases of calcium lead to phosphorus precipitation as calcium phosphates and phosphorus and thus restricting phosphorus availability for the plants. Increasing the potassium uptake in treatments of 2.5 and 5 tons per hectare is due to increased leaf potassium concentration and increased dry matter of leaf yield. The decrease in iron concentration of leaves can be due to the iron precipitation by liming; on the other hand, increasing dry matter yield increases nutrients uptake. It seems that the nearly similar iron uptake in different treatments is due to the interaction between these two factors.

Materials and methods

The paper mill sludge was collected from Pars and Chocka factories, Khoozestan and Guilan provinces, Iran. Total concentrations of some heavy metals (Zn, Cu, Cr, Cd and Pb) in the paper mill sludge were analyzed in the extract after digestion of samples with HNO_3 and HCl (Hossner, 1996). The quantity of the said elements in the digests was determined using inductively coupled plasma atomic emission spectrometry (ICP-AES, LEEMAN LABs, Inc.). The sludge pH and EC (Rhoads, 1996) were determined in a 1:2.5 paper mill sludge/water suspension using a Metrohm 320 pH meter and Metrohm 644 conductometer, respectively. The incubation study was conducted with an acid soil collected from the berry orchards in Rasht, Iran. Soil was air-

Table 5. The effect of treatments on nutrients uptake of soil (g/plot)

Treatment	N	P	K	Fe	Mn	Zn
L_0	8.23 b	17.52 b	17.76 c	0.20 ab	0.15 cd	0.38 c
$L_{2.5}$	14.11 a	24.70 a	39.70 a	0.23 ab	0.25 a	0.93 a
L_5	14.93 a	17.62 b	36.96 a	0.28 a	0.20 b	0.80 a
L_{10}	9.92 b	8.89 c	18.86 c	0.25 ab	0.17 bc	0.57 b
L_C	13.86 a	15.10 b	28.26 b	0.17 b	0.11 d	0.30 c

Table 6. Residual effect of treatments on AB-DTPA-extractable P, K, Fe, Mn and Zn of soil

Treatment	P	K	Fe	Mn	Zn
Lo	14.17 b	157.6 b	88.9 c	44.6 a	1.87 b
L2.5	17.00 b	150.9 b	95.3 c	54.3 a	1.90 b
L5	26.67 a	186.9 a	135.1 b	31.3 b	2.90 a
L10	16.00 b	162.1 ab	244.7 a	44.2 a	0.48 c
Lc	19.30 b	159.8 ab	96.2 c	17.6 c	0.67 c

dried and crushed to pass through a 2-mm sieve. Treatments were then applied to 500g samples of soil and the treated samples were incubated in 1L plastic containers at field capacity (FC) moisture content for up to 90 days. Sub samples were collected after 1, 30 and 90 days of incubation, air-dried and crushed to pass through a 2-mm sieve and stored for chemical analysis. The treatments applied were control, no treatment (L_0); and treated with 0.1, 0.2, 0.5, 1, 2, 4 and 8% W/W dry paper mill sludge ($L_{0.1}$, $L_{0.2}$, $L_{0.5}$, L_1, L_2, L_4 and L_8).

A field experiment was conducted in a berry orchard of the silk worm research center by a randomized completely block design with three replications. Treatments include:

1. Control (no lime),
2. 2.5 ton/ha of paper lime sludge,
3. 5 ton/ha of paper lime sludge,
4. 10 ton/ha of paper lime sludge,
5. 2.5 ton/ha of calcite lime.

Some characteristics of the soils (incubation and field stages) are shown in Table 1. All plots received N-P-K fertilizers, uniformly. After 90 days, leaf dry matter yield was determined after drying of the harvested shoots at $70°C$ for 48 h. Total kjeldahl nitrogen (TKN) of samples were estimated by using a micro-kjeldahl method (Singh and Pradhan, 1981). Subsamples of dry leaf were ground and then dry-ashed in a furnace at $550°C$ and then extracted with 2N HCl. Concentration of Fe, Mn and Zn were measured in the extracts by atomic absorption spectrophotometry, K by flame photometry and P by spectrophotometry (Hossner, 1996). Soil samples from each pot were analyzed for AB-DTPA extractable P, K, Fe, Mn and Zn as well as EC and pH. The samples EC and pH were determined in 1:2.5 soil water suspensions as described before. Data were analyzed by standard ANOVA procedures using MSTATC and SAS softwares and significance were based on $\rho < 0.05$ level for Duncan's Multiple Range Test.

Conclusion

After studying the results of the incubation experiments, it may be concluded that paper sludge lime has good potential in correcting soil acidity by increasing pH in acidic soils. Soil pH modification and subsequent improvement in nutritional status of plants cause an increase in the leaves' dry matter and will lead to uptake of more nutrients. The paper mill sludge amounted to 2.5 and 5 tons per hectare increased the dry matter yield and protein of leaves significantly than in the control sample, so the protein leaves increased in the common lime treatment (calcite). But, taking into consideration dry matter yield, protein and nutrient uptake, jointly, treatments of 2.5 and 5 ha of paper sludge had a better effect in comparison with calcite lime. The results are indicating the potential of paper lime sludge as acidic soil modifiers in the replacement of conventional calcite sources such as calcite which can reduce the disposal costs and possible environmental contamination of this by-product. It is recommended to study the effect of this by-product on the growth of other agricultural products including rice yield and nutrient uptake. Also, in future research the possible contamination of these by-products will be investigated.

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