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INVESTIGATION OF CHEMICAL COMPOSITION AND NUTRITIONAL VALUES OF PULP AND PAPER MILL SLUDGE COMPOST

Pulp and paper industry generates million tons of wastes annually and become one of the major environmental problems worldwide. In recent years, the nutritional values of pulp and paper mill sludge compost attract researchers' attentions. Present study was conducted on paper mill based on used paper. In the present study, distribution trend of the chemical composition and nutritional values of sludge generated from the pulp and paper industry was assessed during composting under different environmental conditions. Aim of the study was to utilize paper mill sludge in environment friendly manner. Standard extraction and analytical methods were employed to test the samples for different parameters including total nitrogen, organic carbon, Electrical Conductivity (EC), pH, phosphate, sulfate, sodium, potassium and four micro-nutrients (Zinc, Copper, Nickel and Chromium). Results demonstrated that many parameters significantly varied during composting and efficiently enhanced the nutritional contents of the compost. The concentration of organic carbon before composting was 8.25 % as compared to the composted sample (> 26 %). An increasing trend of total nitrogen content in the compost was also observed, which revealed the outcome of the ammonification process during composting. In addition, micro-nutrients levels were found in increasing pattern to further improve the nutritional values of the compost. Findings from this study suggested that the nutritional values of sludge significantly improved after amended composting, which could be one of the promising sources of nutrients for enhanced agricultural productivity. Also, the composting process greatly reduced the pathogens in sludge and helps to recycle the important nutrients for enhanced agriculture productivity. It is therefore recommended to use waste of paper mill waste as a rich source of macro and micro nutrients.

Keywords: composting; nutritional values; chemical composition; micro-nutrients; organic carbon.

1. Problem statement and analysis of the recent researches and publications.

Pulp and paper industry is one of the largest industries, playing an integral role in the global economy [1]. Nearly 90 % of pulp and paper is manufactured from wood based materials while the remaining 10 % is manufactured from other kinds of non-wood materials such as bagasse, reeds, rice, straw and wheat straw [2]. This industry is considered to be the major consumer of natural resources (wood, water) and energy in the form of fossil fuels plus electricity [3]. Due to high demand for paper and paper board, the production capacity of this industrial sector is expanding day by day worldwide, generating huge amount of sludge, thus creating serious environmental issues. Safe sludge disposal is a matter of main concern for all the pulp and paper industries [4]. Majority of the sludge is land filled or incinerated in developed countries but it becoming unfavorable disposal option due to financial constraint and environmental pollution [5]. Therefore, it attracts researchers' attention towards some beneficial reuse of paper mill sludge after some pretreatment processes.

Composting could potentially reduce the C: N ratio, odor and volume of the waste produced in paper mill. The prepared compost can suitably be used in horticulture and agriculture [1, 6]. Application of paper mill sludge as soil conditioner is becoming more favorable and environmental-friendly option in present scenario [7]. Previous studies reported that paper mill sludge contains lower concentration of heavy metals and higher concentration of organic matter making the sludge to be reused as valuable source of organic matter for soil [8].

2. Statement of the problem and its solution.

2.1. Purpose of the study.

Pakistan like many other developing countries the demand of paper and paper board steadily increased due to the economic stability. At present nearly 100 small and medium sized paper and paper board mills with the production capacity of about 650 thousand tons per annum are functioning in the country. In addition, Pakistan imports about 36.5 thousands ton of paper and paper board products per annum [9]. But the sludge generated from the pulp and paper industry is mostly dumped in open available areas. During rainy seasons the dumped sludge is carried out by runoff water to the ultimate receiving water body affecting flora and fauna. Thus, the study was designed to investigate the variation of chemical composition and nutritional values of the sludge during composting. The aim of this study was to analyze the sludge collected before and after composting to determine the quantity of various nutrients present. Also, application of different amendments and analyze the prepared compost for macro and micro nutrients' content.

2.2. Materials and methods of investigation

Composting and analysis. The sludge samples from Olmpia Paper and Board Mills, Hayatabad Industrial Estate Peshawar (Pakistan) were collected in plastic containers and analyzed before composting. Sludge samples (before and after composting) were air dried and grounded to pass through a 1 mm stainless sieve and were then stored in the plastic bottles prior to analysis. Paper mill sludge was collected and was composted by blending it with sawdust (organic structural amendment), fruit and vegetable wastes (nitrogenous component). Both, sawdust and kitchen wastes were added manually to the paper mill sludge in different proportions. These mixtures were placed in windrows and were allowed to compost for three months [7]. The compositions of the three compost amendment were made according to the specification:

1. Compost Amendment 1 (CA1): In 1st compost sludge, saw dust and kitchen wastes were mixed in the ratio of 4 : 1 : 1. For this 20 kg of sludge was mixed with 5 kg of each kitchen waste and sawdust.

2. Compost Amendment 2 (CA2): In 2nd amendment sludge, sawdust and kitchen waste were mixed in the ratio of 4 : 2 : 1. For this ratio 17 kg of paper mill sludge were mixed with 8.5 kg of kitchen waste and 4.5 kg of sawdust.

3. Compost Amendment 3 (CA3): In the 3rd compost sludge, sawdust and kitchen waste will be mixed in the ratio of 4 : 3 : 2. For making this ratio 6.7 kg of sawdust 10 kg of kitchen waste was mixed with 13.3 kg of paper mill sludge. The final product obtained was dark brown to black in color with no irritating smell.

These three windrows were watered on every alternate day to maintain the moisture content around 55 %. Windrows were turned over manually with the stick to regulate the oxygen level inside the pile. During early days temperature increased up to 55 – 60 °C later it dropped down to ambient air temperature at the end of process completion.

Compost extraction with Aqua Regia. Compost samples and sub-samples were digested by deploying Aqua Regia digestion method. For this purpose 1 g of powdered samples were taken in digestion tube and 15 mL of aqua regia (3 : 1, $HCl : HNO_3$) was added. The mixture was kept overnight and heated at 80 °C till brown fumes finished to produce. Afterward, it allowed to cool down and 5 mL of $HClO_4$ was added and heated in the fume hood near to dryness or color of the solution becomes white. Extracts obtained were filtered into volumetric flask and diluted upto 50 mL using distilled water [10].

Analytical methods. All the prepared compost samples sub-samples and sample of paper mill sludge before composting were taken, air dried and sieved through 2 mm sieved and extracts were processed for physico-chemical analysis. Each parameter and method of determination is discussed below in detail. The sample pH value was determined by using glass electrode (Model: InoLab pH level-1, WTW, Germany). For this purpose, each sample was mixed with distilled water in the ratio of 1 : 10 sample/water [11]. The compost-water solution was stirred thoroughly and allowed to stand for 30 minutes. After calibrating the pH meter with buffers of pH 7.00, the pH was read by immersing the electrode into the solution and the pH value was recorded [12, 13]. Electrical conductivity was measured of extracts obtained by diluting the samples through distilled water. Saturated solution was made in the ratio of 1 : 10 (compost 10 g: distilled water 90 mL) [11].

Organic carbon in all samples and sub-samples was determined by the modified Walkley-Black method [14]. The process involves a wet combustion of the organic matter with a mixture of potassium dichromate and sulfuric acid. The volume of ferrous ammonium sulfate solution used was recorded and % C calculated [15], as given in equation (1).

$$\%\,C = \frac{(B - S) \cdot M \text{ of } Fe^{2+} \cdot 12 \cdot 100}{\text{Sample weight (g)} \cdot 4000}, \qquad (1)$$

where B and S present the amount Fe^{2+} solution used to titrate blank solution and titrate sample in mL, respectively. In addition, 12/4000 is milli-equivalent weight of C in g, as given in equation (2).

$$\%\,\text{Organic Matter} = \frac{\%\,\text{total C} \cdot 1.72}{0.58}. \qquad (2)$$

Total organic nitrogen was determined by Kjeldahl digestion method. For this purpose, 1 g of air dried grinded samples was taken in digestion tubes and various other chemicals were added in different times according to the instruction manuals [16]. And the absorbance was measured at 450 nm against reagent blank via UV-visible spectrophotometer [17]. For determination of phosphates in the extracts ammonium molybdate ascorbic acid method was deployed and measurement was made by using spectrophotometer at wavelength of 740 µm. The quantity of sodium and potassium was determined by using Flam Photometer (Model: Jenway PF-7, USA). Extracted solution was tested for sulfates by using turbid metric method, in which barium chloride was used to make precipitates of sulfates. Precipitation made the solution turbid; turbidity was measured by using spectrophotometer at wavelength of 425 µm [18]. Total concentration of heavy metals was determined by using aqua regia extraction method [10] and the extract was analyzed on atomic absorption spectrophotometer.

Data analysis and interpretation. Different statistical analysis techniques including descriptive statistics and correlation to interpret the obtained results.

3. Results and discussion

3.1. Variations in different chemical contents during composting.

pH values of soil and all compost samples ranged 7.68 – 8.11, as summarized in table 1. The soil pH was alkaline in nature with pH 8.02. However, variation in pH value was observed after mixing compost materials. Lowest value was recorded in CA1 – 10 % soil with average pH value of 7.92, while highest was observed in CA2 – 15 % and CA2 – 20 % with the average pH value of 8.11. Results showed that pH of all samples were relatively higher as compared to the sludge before composting (table 1).

Table 1 – Summary of pH values of soil and composts

S No	Sample	Min	Max	Average	Std. D
1	Sludge before compost	7.18	8.18	7.68	0.34
2	Soil (100 %)	7.62	8.42	8.02	0.27
3	CA1	7.53	8.53	8.03	0.34
4	CA2	7.44	8.44	7.94	0.34
5	CA3	7.15	8.15	7.65	0.34
6	CA1-10%	7.42	8.42	7.92	0.34
7	CA1-15%	7.47	8.47	7.97	0.34
8	CA1-20%	7.48	8.48	7.98	0.34
9	CA2-10%	7.59	8.59	8.09	0.34
10	CA2-15%	7.61	8.61	8.11	0.34
11	CA2-20%	7.61	8.61	8.11	0.34
12	CA3-10%	7.42	8.42	7.92	0.34
13	CA3-15%	7.48	8.48	7.98	0.34
14	CA3-20%	7.51	8.51	8.01	0.34

Generally, high pH values make the compost samples alkaline in nature and paper mill sludge compost may neutralize acidic soils for better crop. Variation of compost pH indicates the process decomposition and stabilization and it drops initially, but later on it suddenly increase up to the range 8 – 8.5 due to ammonification process. Generally, compost stabilizes at pH 8 [19] and the maximum acceptable range of pH range 5.5 – 9 in good quality compost [20]. In this study, the pH of soil and compost recorded was moderately alkaline to strongly alkaline and can be considered as a good soil conditioner for acidic soil.

In the present study, EC values ranged from 152.03 µS/cm (CA1) to 988.65 µS/cm sludge sample before composting (table 2). General range of EC range 200 – 1200 µS/cm for agricultural soil. Lower EC (< 200 µS/cm) demonstrates of lower plant nutrients availability while above 1200 µS/cm may lead to saline conditions [21]. In compost low conductivity is desirable as it indicates the presence of complex nutrients. Decrease in EC in composting process occurs due to the increase in nutrient concentration such as nitrates and nitrites. Finished compost may show quite lower values of EC than that of the raw material [19]. EC values of the present study revealed significant variations between the initial and after composting EC values of paper mill sludge. However, the EC values were within the maximum permissible limit for agriculture soil i.e. 4000 µS/cm [22].

Table 2 – Summary of EC (µS/cm) values of soil and composts

S. No	Sample ID	Min	Max	Average	Std.D
1	Before composting	987.30	990.00	988.65	0.97
2	CA1	149.83	154.10	152.03	1.47
3	CA2	153.50	158.50	155.70	1.88
4	CA3	155.70	156.50	156.10	0.29
5	CA1-10%	207.00	207.50	207.25	0.18
6	CA1-15%	309.60	310.50	310.05	0.32
7	CA1-20%	412.40	414.00	413.20	0.58
8	CA2-10%	224.00	226.20	225.10	0.79
9	CA2-15%	337.80	339.00	338.40	0.43
10	CA2-20%	441.60	452.00	446.80	3.74
11	CA3-10%	222.00	224.30	223.15	0.83
12	CA3-15%	332.10	333.00	332.55	0.32
13	CA3-20%	442.80	444.00	443.40	0.43
14	100 % Soil	225.40	227.00	226.20	0.58

Figure 1a present the composition of total nitrogen during composting. Soil organic carbon and nitrogen are the most important factors in determining soil fertility. Results revealed of highest total nitrogen value (0.35 %) in CA3 – 20 % sample, while lowest value (0.08 %) was recorded in the sludge sample before composting, as shown in figure 1a. It can be seen that the values in pure compost samples (CA1, CA2 and CA3) were 0.11, 0.14 and 0.22 %, respectively. The content of total nitrogen in CA1 treated soil (10, 15 and 20 %) was 0.18, 0.2 and 0.23%, respectively. Relatively high nitrogen content in CA3 treated soil may be attributed to the addition of highest amount of kitchen waste (initial ingredient) in CA3 compost. Total nitrogen is the sum of organic and inorganic nitrogen present in the compost and the inorganic nitrogen is present in the form of ammonium and nitrate nitrogen. However, from research in [20] concluded that the addition of amendment could increase corn yield but had no significant impact on concentration of nutrients organic matter and pH of soil.

The value of phosphates before composting was observed to be about 1.11 mg/kg while concentration of phosphates recorded in soil was 0.67 mg/kg. Sludge composting slightly enhanced the level of phosphorus compounds and the values observed in pure compost samples (CA1, CA2 and CA3) were 0.98, 1.80 and 1.13 mg/kg, respectively, as shown in figure 1b. The values observed in CA1 treated soil samples (CA1 – 10, 15 and 20 %) were 0.68, 0.70 and 0.73 mg/kg, respectively, while in CA2 treated soil samples (CA2 – 10, 15 and 20 %) values of phosphates observed were 0.78, 0.82 and 0.90 mg/kg, respectively (figure 1b).

Figure 1c present the results of potassium during composting. Results obtained from the present study showed that sludge sample before compost contained 1035 mg/kg potassium, while pure soil has 945 mg/kg potassium. Pure compost samples CA1, CA2 and CA3 were observed K values of 1638, 1820 and 2025 mg/kg, respectively (figure 1c). Slightly increasing trend in level of K was observed after composting, as shown in figure 1c. This increase may be attributed to the usage of fruit and vegetable wastes as initial ingredient in compost. The recommended value of K in good quality compost ranges 6000 – 17000 mg/kg [24] and the pure compost samples were deficient of K.

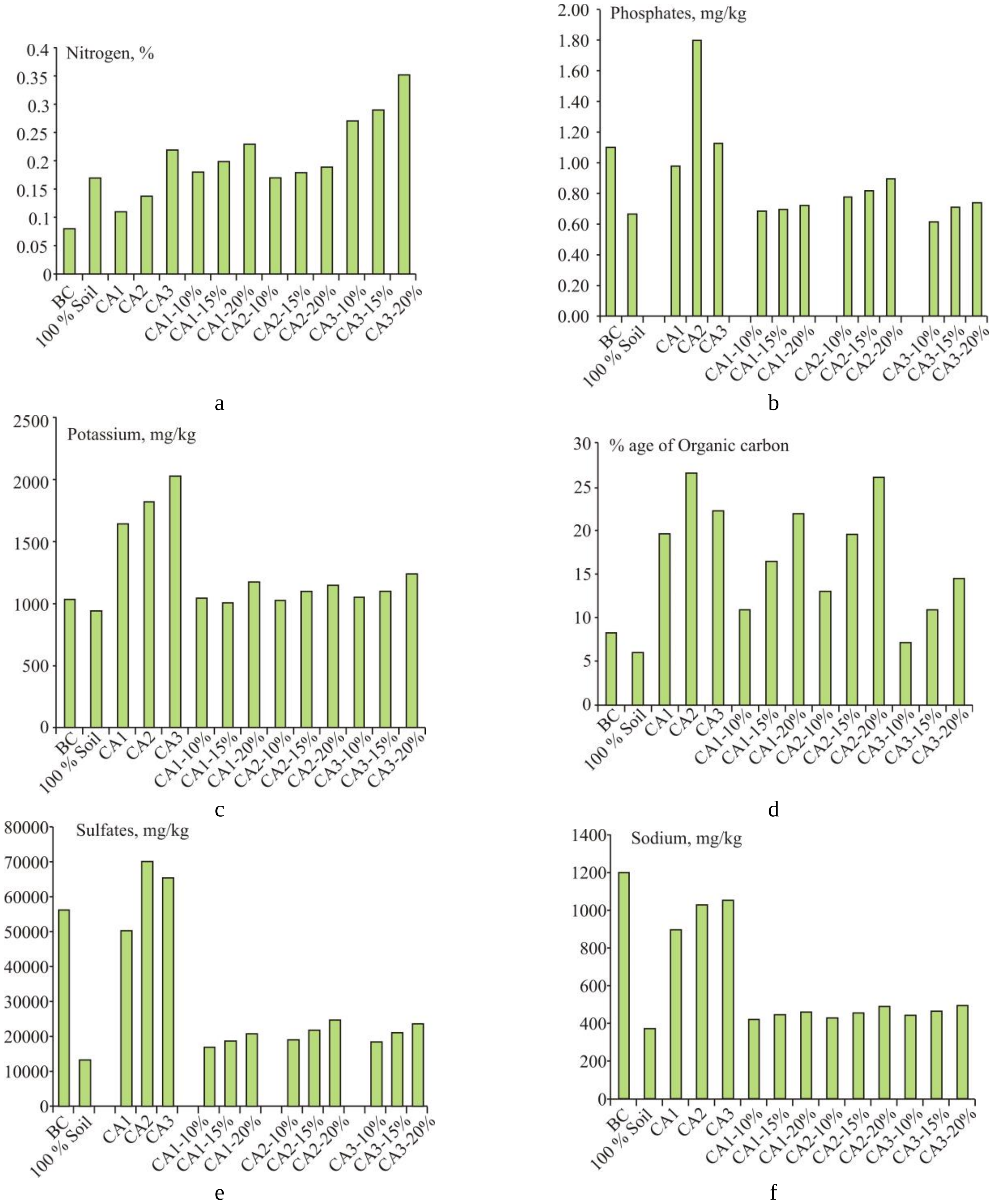

Figure 1 – Concentrations of elements in compost and treated soil samples: a – of total nitrogen; b – of phosphate; c – of potassium; d – of organic carbon; e – of sulfate; f – of sodium

The highest organic carbon (C) content of 26.55 % was observed in CA2 pure compost sample while lowest percentage of 6 % was recorded in 100 % soil, as shown in figure 1d. The concentration of organic C in sludge before composting (8.25 %) was found low as compared to compost samples. In the three compost samples (CA1, CA2 and CA3) the values of organic C recorded were 19.5, 26.55 and 22.2 %, respectively. In CA1 treated soil (10, 15 and 20 %) the organic C content recorded was 10.95, 16.42 and 21.9 %, respectively (figure 1d). An increasing trend of organic C was observed while amendment from 10 % to 20 % samples and can be clearly seen in figure 1d. Organic amendments generally increases organic matter content of the soil which in turn enhances water holding capacity, aeration, aggregating property and microbial community structure [25]. Organic matter of soil has strong tendency to adsorb essential metals from soil and make it available to plant [26]. The organic matter in all the composts as well as treated soil samples calculated was found enough according to the international standards for agricultural soil.

In sludge sample before composting sulfates was found to be about 56344 mg/kg, as shown in figure 1e. Highest sulfates content was determined in pure compost sample CA2 (70053 mg/kg) while lowest was observed in soil sample was found to be about 13325 mg/kg (figure 1e). In CA3 treated soil samples (10, 15 and 20 %) values of sulfates were 18568, 21176 and 23792 mg/kg, respectively (figure 1e).

Figure 1f shows trend of varying Na concentration in all treated soil samples. Sodium (Na) is not considered as essential nutrient required for plants but its concentration effects soil structure. Presence of high concentration of Na may cause adverse impacts on growth of plants and physical and chemical properties of soil. In present study, the concentration of sodium ranges 1200 – 370 mg/kg in all the samples. Highest level was recorded in sample sludge before composting (1200 mg/kg) while lowest was 370 mg/kg in soil sample (figure 1f). In all treated soil samples concentration of Na was quite in normal range.

3.2. Essential micro-nutrients' variation during composting.

There are many bio-essential nutrient elements required to plants and animals in trace quantities to maintain essential body functions. In the present study, four micronutrients were focused to be studied different samples (soil, sludge and compost).

Zn is one of the important trace elements for plants, animals and human beings. It is needed for plants in various enzymatic activities, oxidation-reduction reactions as well as in metabolic processes [27]. Sludge sample before composting contained highest content of zinc (255 mg/kg), but once it was subjected to composting along with sawdust and kitchen waste its concentration declined, as shown in figure 2a. For instance, the concentrations were 213, 207.4 in CA1 and CA2, respectively. The values of Zn recorded in various amendments were 80.5, 87.5, 95.2 mg/kg in CA1 – 10, CA1 – 15 and CA1 – 20 %, respectively (figure 2a). Similarly, Zn concentration in CA2 – 10 %, 15 % and 20 % was recorded as 81.8, 89.3 and 96.6 mg/kg, respectively. All Zn values were within the maximum concentration of metals in the good quality compost. However, Zn content was well below maximum toxic level of 700 mg/kg for metals in compost reported earlier [22]. Similarly, Zn values in the treated soil samples were observed in the same level (87.6 mg/kg) from a previous study reported from China [28].

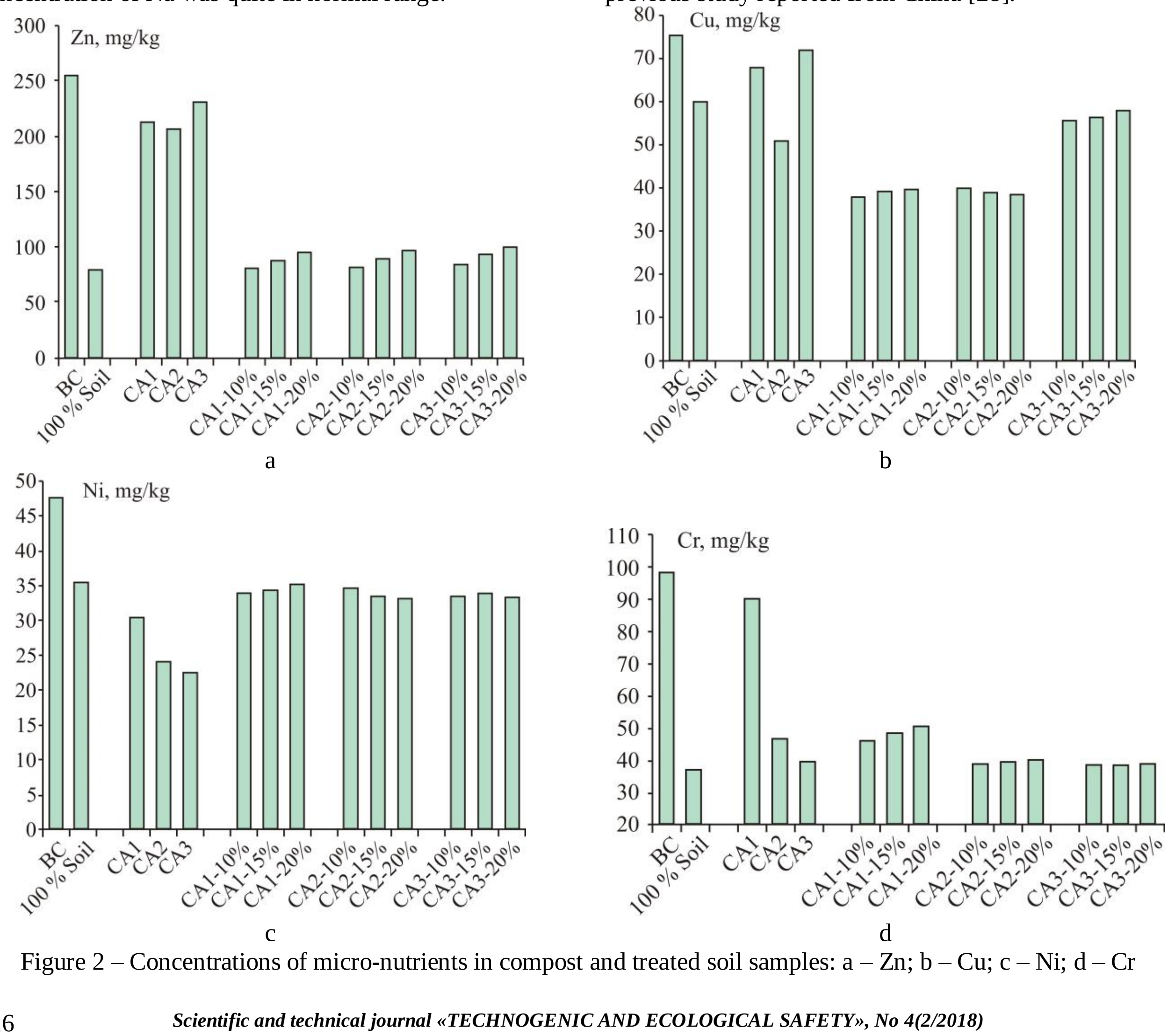

Figure 2 – Concentrations of micro-nutrients in compost and treated soil samples: a – Zn; b – Cu; c – Ni; d – Cr

Figure 2b presents the distribution trend of Cu in compost and treated soil samples. Cu value in sludge sample before composting was found 75.4 mg/kg while the maximum level in all other samples was recorded 72.1 mg/kg. However, low concentration (60 mg/kg) was recorded in soil sample (figure 2b). A gradual increased Cu level was observed with the increasing amount of compost in CA1 as well as in CA3 treated soil samples, as shown in the figure 2b. Results findings revealed that the Cu level in all treated soil samples was below the maximum permissible limit for soil 135 – 270 mg/kg and also for agriculture soil set [22].

Figure 2c presents the distribution trend Ni in compost and other samples. Results demonstrated that highest concentration of Ni was found 47.6 mg/kg in sludge before composting while lowest was observed (22.4 mg/kg) in CA3 sample. In addition, the Ni values in CA3 – 10, 15 and 20 % were 33.4, 33.8 and 33.2 mg/kg, respectively (figure 2c). The maximum desirable level of Ni in unpolluted soil was reported to be about 35 mg/kg [26]. The concentration of Ni in the treated soil samples was found within the maximum allowed range of trace elements in soil (US-EPA 1995).

Figure 2d presents the concentration of Cr recorded in compost and other treated soil samples. Results showed that the highest Cr level in sludge before composting was recorded 98.3 mg/kg while lowest value was recorded in CA3 sample (39.3 mg/kg). In all the treated samples the value of Cr showed an increasing trend, as shown in figure 2d. For instance, Cr value in sample CA2 for 10, 15 and 20 % values observed were 37.7, 39.3 and 40 mg/kg, respectively. Anthropogenic activities may enhance Cr content in the soil [29].

Conclusions.

In the present study, variation of the chemical composition and nutritional values of the compost was investigated. Results demonstrated that all the compost samples were found rich in organic carbon content and alkaline pH values, which can be considered to enhance the buffering capacity of soil. EC ranged (207.25 – 446.80 µS/cm) in all the samples. Two major macro nutrients (total nitrogen and phosphate) were found lower than the desired level. However, potassium level in soil after treatment with compost was found significant. Micro-nutrients (Zn, Cu, Cr and Co) levels were found higher in compost samples but did not exceed the maximum permissible limits. Findings from this study suggested that the optimized composting process can improve the nutritional values and could be a promising source of nutrients for enhance agricultural productivity.

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Conflicts of Interest.

None of the authors have any potential conflicts of interest associated with this present study.

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ДОСЛІДЖЕННЯ ХІМІЧНОГО СКЛАДУ І ПОЖИВНОЇ ЦІННОСТІ КОМПОСТУ З ОСАДУ ЦЕЛЮЛОЗНО-ПАПЕРОВОЇ ФАБРИКИ

Целюлозно-паперова промисловість щорічно виділяє мільйони тонн відходів і стає однією з основних екологічних проблем в усьому світі. В останні роки поживна цінність осаду целюлозно-паперового комбінату привертає увагу дослідників. Це дослідження проводилося на основі відходів, утворених на целюлозно-паперовій фабриці. У дослідженні оцінена тенденція розподілу хімічного складу і поживних речовин осаду, що утворюється в целюлозно-паперовій промисловості, при компостуванні в різних умовах навколишнього середовища. Мета дослідження полягала в тому, щоб використовувати осад паперової фабрики в екологічно чистому вигляді. Для перевірки зразків з різними параметрами, включаючи загальний азот, органічний вуглець, електропровідність, pH, фосфат, сульфат, натрій, калій і чотири види мікроелементів (цинк, мідь, нікель і хром), використовували стандартні екстракційні і аналітичні методи. Результати показали, що під час компостування багато параметрів істотно змінювалися і ефективно покращували поживну цінність компосту. Концентрація органічного вуглецю перед компостуванням становила 8,25 % у порівнянні із зразком, що компостувався (> 26 %). Спостерігалася також тенденція до збільшення загального вмісту азоту в компості, що виявило результати процесу аммонификации при компостуванні. Крім того, рівні мікроелементів змінювалися за зростаючою схемою для подальшого поліпшення поживної цінності компосту. Висновки з цього дослідження показали, що поживні цінності осаду значно покращилися після внесення поправок в компостування, що може стати одним з перспективних джерел поживних речовин для підвищення продуктивності сільського господарства. Крім того, процес компостування значно зменшив кількість патогенних мікроорганізмів в осаді і сприяв переробці важливих поживних речовин для підвищення продуктивності сільського господарства. Тому рекомендується використовувати відходи паперової фабрики як багате джерело макро- і мікроелементів.

Ключові слова: компостування; поживна цінність; хімічний склад; мікроелементні; органічний вуглець.

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ИССЛЕДОВАНИЕ ХИМИЧЕСКОГО СОСТАВА И ПИТАТЕЛЬНОЙ ЦЕННОСТИ КОМПОСТА ИЗ ОСАДКА ЦЕЛЮЛОЗНО-БУМАЖНОЙ ФАБРИКИ

Целлюлозно-бумажная промышленность ежегодно выделяет миллионы тонн отходов и становится одной из основных экологических проблем во всем мире. В последние годы питательная ценность осадка целлюлозно-бумажного комбината привлекает внимание исследователей. Настоящее исследование проводилось на основе отходов, образованных на целлюлозно-бумажной фабрике. В исследовании оценена тенденция распределения химического состава и питательных веществ осадка, образующегося в целлюлозно-бумажной промышленности, при компостировании в различных условиях окружающей среды. Цель исследования состояла в том, чтобы использовать осадок бумажной фабрики в экологически чистом виде. Для проверки образцов с различными параметрами, включая общий азот, органический углерод, электропроводность, pH, фосфат, сульфат, натрий, калий и четыре вида микроэлементов (цинк, медь, никель и хром), использовали стандартные экстракционные и аналитические методы. Результаты показали, что во время компостирования многие параметры значительно изменялись и эффективно улучшали питательную ценность компоста. Концентрация органического углерода перед компостированием составляла 8,25 % по сравнению с образцом, который компостировали (> 26 %). Наблюдалась также тенденция к увеличению общего содержания азота в компосте, что выявило результаты процесса аммонификации при компостировании. Кроме того, уровни микроэлементов изменялись по возрастающей схеме для дальнейшего улучшения питательной ценности компоста. Выводы из этого исследования показали, что пищевая ценность осадка значительно улучшилась после внесения поправок в компостирование, что может стать одним из перспективных источников питательных веществ для повышения продуктивности сельского хозяйства. Кроме того, процесс компостирования значительно уменьшил количество патогенных микроорганизмов в осадке и способствовал переработке важных питательных веществ для повышения продуктивности сельского хозяйства. Поэтому рекомендуется использовать отходы бумажной фабрики как богатый источник макро- и микроэлементов.

Ключевые слова: компостирование; питательная ценность; химический состав; микроэлементные; органический углерод.