Organic Biofertilizer from Brewery Wastewater Sludges via Aerobic Composting Process

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Abstract. The main purpose of this research was investigating the potential of aerobic composting process for Preparation of biofertilizer from brewery wastewater sludge (BWS) and spent brewery diatomaceous sludges (BSDS). In this study, BWS was mixed with BSDS sludges and, yielding three different mixtures to be composted. The composting process was assessed through measurements of temperature, moisture, pH, electrical conductivity, organic carbon, organic nitrogen, and total organic phosphorus contents. Moreover, the total concentration of heavy metals (Cr, Cu, Cd, Hg, and Pb) and the plant nutrients (Na, K, Mg, Ca, and Fe) were determined. Additionally, the degradation degree was evaluated through the detection and quantification of E. coli, Salmonella, and Fecal Coliform. It was found that the ratio of C/N was 9:1 and phosphorus fluctuated around 8.5%. Cr, Cu, Cd, Hg, and Pb were found 1.95 ± 0.045, 5.36 ± 0.03, 0.475 ± 0.004, 0.165 ± 0.004 and 0.273 ± 0.005 mg/kg respectively. Plant Nutrients Na, K, Mg, Ca and Fe were found at a concentration level of 100.16 g/kg, 122.95, 869.92, 4084.08 and 26.86 mg/kg respectively. Generally, aerobic compost of BWS and BSDS yielded acceptable quality of biofertilizer in line with EPA standard value limits. The higher pathogen (E. coli and Fecal Coliform) contents of raw sludges are stabilized in composting to the required standards and found far below upper EPA standard limits for unrestricted agricultural application.

Keywords: Aerobic composting · Fertilizer · Kieselguhr sludges · Brewery wastewater sludges · Metal contents

1 Introduction

The brewing industry is one of the largest water users industries and approximately 3–10 L of waste effluent is generated per litre of beer [1]. Breweries in Ethiopia have the potential to produce more than 12 million hectoliters of beer per year [2]. The high organic content of brewery effluent classifies it as a very high strength waste 1000–4000 mg/L COD and 1000–1500 mg/L BOD [3, 4]. Due to increasing environmental concerns and regulations, attempts are to utilize this brewery byproduct in an environmentally friendly manner [3, 5]. In developing countries, non-utilization of byproducts is a drag on economic growth in addition to their environmental burden [5].

N. G. Habtu et al. (Eds.): ICAST 2019, LNICST 308, pp. 3–15, 2020.
https://doi.org/10.1007/978-3-030-43690-2_1

Since all Ethiopian breweries use diatomite for clarification, the total amount of brewery spent diatomite sludge (BSDS) produced, each year is estimated to be about 69,000 metric tons [6]. Diatomite waste is a major challenge for all breweries due to its economic and environmental consequences [5]. Consequently, BSDS is often dumped in landfills that releases leachate to ground water and carbon monoxide as well as carbon dioxide to the atmosphere. This contributes to global climate change and promotes growth of microbes [7]. Moreover, the high moisture content of BSDS (approximately 70%) and its chemical composition lead to rapid degradation, so that the open dumping produces unpleasant odors [8]. Although chemical analysis of spent diatomaceous earth has high nutrient content, particularly of organic nitrogen [9], the environmental risks associated with its application are not well known. Moreover, nitrate production from BSDS is released slowly; hence, it has a lower leaching risk and advantages for crop production [10]. Waste Management and disposal by composting is one of the most promising technologies for treating biosolids allowing their recycling. Brewery sludge originates from the food industry is rich in nutrients and organic matter, so it can be used to produce a high-quality organic compost. This reduces environmental impact and increases crop productivity [11]. Waste sludges generated from Dashen Brewery Share Company was given to farmers for crop production. However, due to its bad odors and other problems, the farmers stopped using sludges and nowadays it is discharged as landfill. Thus, this paper investigates the suitability of brewery wastewater sludge through an aerobic composting as organic fertilizer for agriculture application.

2 Materials and Methods

2.1 Reagents and Chemicals

Analytical grade Ammonium Molybdate, Ammonium Meta Vanadate, Orthophosphate (KH_2PO_4), Hydrochloric acid (HCl 36%) were purchased from Addis Ababa. Concentrated Sulfuric acid (H_2SO_4 98%), Hydrochloric acid (HCl 37%), Potassium sulphate (K_2SO_4 99%), Copper sulphate ($CuSO_4.10H_2O$), Boric acid (H_3BO_3 99.5%), Sodium hydroxide (NaOH 98%), Bromcresol green and Methyl red indicator were used in nitrogen determination. In plant nutrients determination, Acetic acid (36%) and Ammonium hydroxide (NH_4OH 30%) were used. Distilled and deionized water were used to prepare the mother solution.

2.2 Experimental Setup and Description

The aerobic composting reactors (Fig. 1) are connected to cold-water tank and air pipe (perforated part in composters). The composting materials such as brewery wastewater sludges (BSW), brewery spent diatomaceous sludges (BSDS), cow dung and tap water were fed to the aerobic batch composter. It was well mixed and aerated.

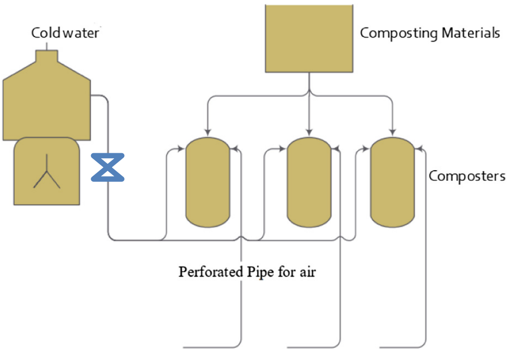

Fig. 1. Experimental setup

2.3 Raw Sludges Preparation and Composting

Dry Brewery wastewater sludges (BWS) and brewery spent diatomaceous sludge (BSDS) were collected from Dashen brewery Share Company and ground by Roller Mill (D-55743 Idar-Oberstein) and sieved through a 1.8 mm sieve. Samples were weighed by digital balance (CAS-164) and slurry was prepared using tap water for composting. The composting experiment is designed using full factorial design i.e. two factors (brewery sludges ratio and moisture content) and three levels with replicate. In composting when the BWS to BSDS ratio is lower than 0.50 the microbial activity is hindered due to lack of enough food. As a result, in order to breakdown the waste, the ratio was maintained between 0.50 and 0.95. The composting sludge substrate contains moisture content of 60–80% [12]. If moisture falls below 40%, microbial activity will slow down. If the moisture exceeds 80%, aeration is hindered, and anaerobic decomposition appeared [12]. Experiments were conducted using mixtures of milled BWS and BSDS in a batch plastic composters (1.5 kg) at different ratios and moisture contents over 30 days. The composting material was manually stirred three times each week to ensure proper mixing and aeration. Compost samples were collected from composters to analyze the physicochemical parameters.

2.4 Characterization and Analysis

2.4.1 Determining Organic Matter and Water Holding Capacity (WHC)

Volatile solids were determined by the loss-on-ignition (LOI) method. The 25 g sample was put in a weighed crucible and was dried in oven dryer (DHG-9140) for 24 h at 105 °C. It was cooled in desiccators and it was reweighed and then ignited in the furnace (AF-11/16) at 500 °C for 30 min. Percentage of organic matter (OM) was calculated using the following equations.

$$\text{Organic Matter } (\%) = \frac{wd - wa}{wd} \times 100\% \quad (1)$$

Where Wd = dry weight sample and Wa = ash weight after combustion.

The organic carbon was determined using empirical equation developed by Pribyl [13]. The percentage of carbon was estimated satisfactorily from the percentage of organic matter by the Eq. (2)

$$\text{Organic Carbon }(\%) = \frac{Organic\,matter(\%)}{1.8} \tag{2}$$

In water holding capacity (WHC) determination, a dried 100 g sample was added in funnel covered with filter paper and 100 mL tap water was poured. Amount of water added and drained was recorded. Then the quantity of water retained in 100 g sample was calculated.

$$\text{WHC }(\%) = \frac{\text{Water Added (mL)} - \text{Water Drained (mL)}}{\text{Water Added (mL)}} * 100\% \tag{3}$$

2.4.2 Determination of Organic Phosphorous and Total Organic Nitrogen

The amount of organic Phosphorous in the sample was determined by UV-Vis spectrophotometer (Cary 60) at a wavelength (λ) of 410 nm according to the method described in APHA (1999).

Nitrogen was determined by Kjeldahl method. A 1 g sample was placed in Kjeldahl flask digester (DK-20) and 15 mL concentrated sulfuric acid was added. A 1 g catalyst (mixture of copper sulphate and potassium sulphate) was added to the flask and it was heated. The solution then was distilled in distiller (UDK-169) with 40% NaOH solution that can convert the ammonium salt to ammonia. The amount of ammonia present in the sample was determined by titrating it with 0.1 N HCl in boric acid. The boric acid captures the ammonia gas. Finally, the amount of nitrogen in a sample can be calculated from the quantified amount of ammonia ions in the receiving boric acid solution (APHA, 1999).

2.4.3 Metal Analyses Using Di-Acid and Ammonium Acetate Method

A Di-Acid (HNO_3-$HClO_4$) method was used to determine metal contents. A 1 g air-dry sample (0.15 mm) was added into 300 mL calibrated tube. Then 3 mL concentrated HNO_3 was added and swirled carefully. The temperature setting was slowly increased to 145 °C for 1 h. Then, 4 mL of concentrated $HClO_4$ was added and heated it to 240 °C for further 1 h. Then the tubes were cooled to room temperature, and filtered through Whatman No. 42 filter paper, and brought to 50 mL volume. Each batch has one reagent blank (without samples). Finally, the metals were determined through Atomic Absorption Spectrophotometer (NOV400P).

In Ammonium Acetate Solution preparation, 700 mL of distilled water, 57 mL acetic acid and 68 mL ammonium hydroxide were added and mixed together. The solution was stirred using magnetic stirrer (MS7-H550-Pro) and its pH was adjusted to pH 7 using droplet acetic acid or ammonium hydroxide. The solution was then transferred to a 1.0 L volumetric flask and top up to the mark with distilled water. 4 g air-dried and milled sample was put in a plastic centrifuge tube. 33 mL of ammonium acetate solution was added and was shaken in a mechanical shaker (@Heidolph Unimax 2010) for 1 h. Then, the tube was centrifuged and clear supernatant was decanted into 100 mL volumetric flask and top-up to the mark with distilled water. Finally, metals were determined through Photometer (7100 Palintest).

2.4.4 Microbiological Analysis

Using Eosin Methylene blue agar (modified) Levine: This versatile medium, modified by Levine (Levine M., 1921) was used for the differentiation of Escherichia coli and Enterobacteria aerogenes. Medium was prepared to the formula specified by the APHA for detection and differentiation of coliform group of organisms (10 g peptone, 10 g lactose, 2 g Dipotassium hydrogen phosphate, 0.4 g Eosin Y, 0.065 Methylene blue, 15 g agar at pH 6.8). The sum of these medium types 37.5 g was dissolved in 1 L of distilled water. It was brought to the boil to dissolve completely and which was sterilized by autoclaving at 121 °C for 15 min. Then it was cooled to 60 °C and the medium was shaken in order to oxidize the methylene blue and to suspend the precipitate, which is an essential part of the medium. Using Brilliant green bile (2%) broth: the medium is recommended for the 44 °C confirmatory test for Escherichia coli. Sum (10 g peptone, 10 g lactose, 20 g ox bile (purified), 0.0133 g brilliant green, 0.065 Methylene blue, 15 g agar at pH 7.4) weight of 40.0783 g was dissolved in 1 L of distilled water. It was mixed well and it was distributed into containers fitted with tubes and sterilized by autoclaving at 121 °C for 15 min. To indicate the presence of Escherichia coli, Brilliant Green Bile Broth is incubated at 44 ± 1 °C for 48 h. Standard plate count (SPC) method: 23.5 g Plate Count Agar was diluted in 1 L distilled water. Serial dilution: A solution was prepared from 9.5 g Maximum recovery diluents and 1 L distilled water, which was used to dissolve the sample.

2.4.5 Statistical Analysis

IBMS Statistics 20 statistical software was used in this study for all statistical analysis. Two Way ANOVA was used for testing whether there was any significant difference in compost quality among compost profiles for two factors (moisture content and brewery sludge ratio) and three levels of factors at 5% level of probability.

3 Results and Discussion

3.1 Organic Nutrients Analysis of Raw Sludges

The analysis of selected nutrients in brewery wastewater sludge (BWS) and brewery spent diatomaceous sludge (BSDS) is shown in Table 1. These results revealed that the sludge is rich in organic nitrogen, phosphorous, organic matter, organic carbon, high water holding capacity (WHC) and high conductivity. These sludges have high total dissolved solid (TDS) content, but it contained very low salinity.

With respect to pH, the sludges contained pH of 8.13 ± 0.07 BWS and 7.71 ± 0.06 BSDS indicating slightly basic properties and previous studied show 6.5–11.5 pH of brewery sludges [14]. These results indicated that the two sludges could be used as organic fertilizer with proper treatments.

Table 1. Raw brewery and Kieselguhr sludges physicochemical characteristics

Parameters	BWS	BSDS
	Value	Value
Organic nitrogen %	4.79 ± 0.05	2.74 ± 0.04
Organic phosphorous %	8.29 ± 0.26	4.03 ± 0.04
Organic matter %	53.25 ± 0.43	28.28 ± 0.23
Organic carbon %	33.28 ± 0.27	17.68 ± 0.14
WHC (%)	66.66 ± 0.54	40.4 ± 0.33
Conductivity (μS/cm)	803.96 ± 6.5	424.91 ± 3.43
TDS (mg/L)	286.89 ± 2.32	279.67 ± 2.26
Salinity	0.293 ± 0.002	0.293 ± 0.003
pH	8.13 ± 0.07	7.71 ± 0.06

3.2 Metal Analysis and Pathogen Determination of Raw Sludges

As it can be seen in Table 2, characterization results revealed that primary nutrients (N, P, K) and secondary nutrients with high organic matter content and high water holding capacity indicate that brewery sludges is a potential source of organic fertilizer. As it can be seen in Table 2, the heavy metal concentrations in BWS are higher than threshold limit [16]. The sources of heavy metals in BWS is disinfection agents, cleaning detergents, antifungal paste solution, residual furnace oil, chemicals in beer quality analysis laboratory, residual printer inks (Fe), paper ash (Cd and Pb) [17] and toilet sludges (feces and urine). But, heavy metal content in BSDS is below the threshold limit and it has a high silicon dioxide content exceeding 33.51 ± 0.065% that can scavenge [18] heavy metals ions and reduce heavy metal contents in composting process since they are non-biodegradable and toxicity nature once they are present [18, 19].

The concentrations of total fecal coliforms (TFC) and Escherichia coli (E.coli) as dry weight in BWS and BSDS were determined. In BWS it was found 2031 CFU/g TFC and 160 CFU/g E.coli, which are higher than the EPA value 100 CFU/g. Similarly, total fecal coliforms (TFC) and E.coli as dry weight in the BSDS samples were 65 CFU/g and 55 CFU/g respectively, which were lower than the EPA 100 CFU/g. However, the pathogen content in BWS was high which needs further treatment.

Table 2. Composition of Raw BWS and BSDS sludges

Metals	BWS	BSDS	Limit [28, 29]
Copper, Cu (mg/kg)	6.28 ± 0.04	0.97 ± 0.01	70
Iron, Fe (mg/kg)	25.56 ± 3.52	26.03 ± 3.25	266
Sodium, Na (mg/kg)	112.2 ± 0.91	58.83 ± 1.84	214
Magnesium, Mg (mg/kg)	1126.93 ± 2.59	838 ± 34.29	1478
Calcium, Ca (mg/kg)	3433.79 ± 15.45	3118.5 ± 5.31	5789
Potassium, K (mg/kg)	116.95 ± 0.48	119.2 ± 2.29	6108
Lead, Pb (mg/kg)	10.627 ± 0.65	0.205 ± 0.012	45
Cadmium, Cd (mg/kg)	1.633 ± 0.025	0.305 ± 0.02	0.70
Mercury, Hg (mg/kg)	0.82 ± 0.21	0.215 ± 0.004	0.70
Chromium, Cr (mg/kg)	31.5 ± 0.24	0.89 ± 0.041	25
Silicon oxide (SiO2) (%)		33.51 ± 0.065	

3.3 Compost Product Characterization

The pH of final compost was measured by pH (BANTE 90-UK) meter and was found between 7.5–7.63 that indicates alkaline properties and similar results were observed in previous studies [3]. The electrical conductivity (EC) values of compost samples were measured (YSI Pro 30) and found to be 900–1650 μS/cm that is far below the permissible limit (4000 μS/cm). The extraction of total dissolved solid (TDS) is influenced by moisture content of composting substrates and found 500–1100 mg/L which is far above the control soil (3.33 mg/L). Organic matter was fluctuated in all treatments. It ranges between 41–48% of organic matter and 28–30% organic carbon. This fluctuation points out the mineralization and degradation of organic matter [20].

3.4 Organic Nitrogen

The nitrogen content of compost samples from 50%, 60%, 70% and 80% moisture content show different trends as the BWS ratio was increased as it is illustrated in Fig. 2. In 70% moisture content, the results revealed that organic nitrogen was increased as the BWS ratio is became high and then it was decreased due to volatilization of ammonia [22]. When the moisture content is increased to 80% the organic nitrogen is decreased due to the creation of anaerobic conditions for microorganisms that they cannot completely decompose biodegradable content of composting materials. Since aeration is hindered, nutrients are leached out and decomposition slows down. Organic nitrogen content of 50% moisture content is lower than the 60%, 70% moisture contents experiments because microbial growth and activity was hindered by lower moisture content. Nitrogen content is significantly different at various moisture content and brewery wastewater sludges (BWS) ratio ($P < 0.05$) because the brewery sludge is abundant in organic nitrate came from residue barley malts, hot trub and others [17].

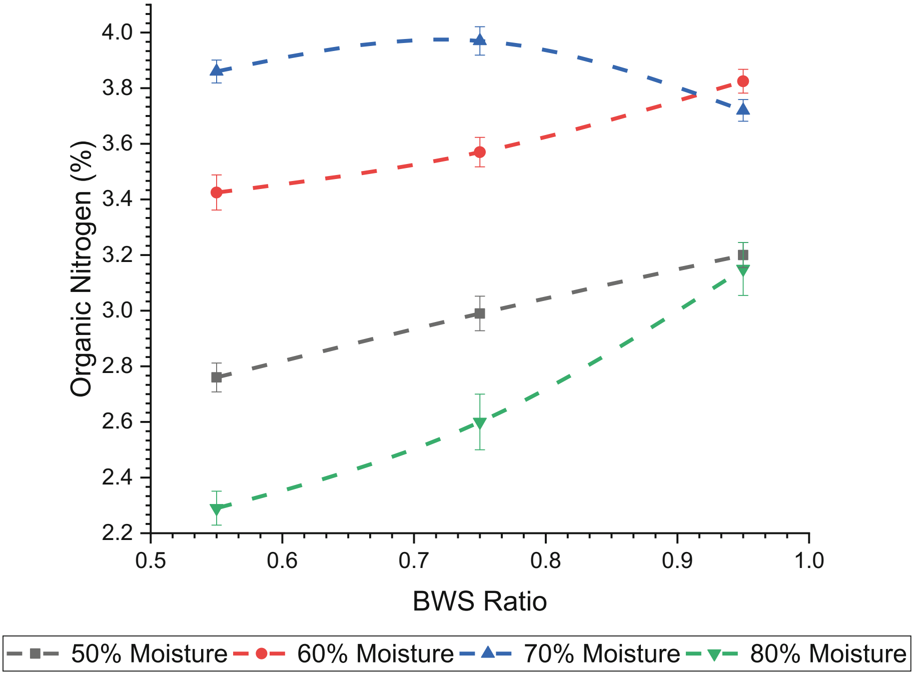

Fig. 2. Organic nitrogen percentage of final compost as function of BWS ratio

3.5 Total Organic Phosphorus

In compost samples, the maximum concentration of total phosphorus (9.5%) was found at 0.95 BWS ratio and 70% moisture content in the compost samples, while the minimum phosphorus was found between 0.55 and 0.95 BSW ratio and 80% moisture content, as illustrated in Fig. 3. As shown in Fig. 3, phosphorus percentage of the compost increases as BWS increases since the BWS and BSDS sludge are naturally rich in phosphate compounds [17]. During composting process, phosphorus does not experience volatilization as nitrogen. Phosphorus settles in bacterial cells and mixed with composts, phosphorus is the sum of dissolved and insoluble forms [24]. As a result, phosphorous content of compost is increased with increasing BWS ratio. In previous studies, similar findings were found in the final composts [23]. Phosphorus content was significantly different at various moisture and BWS ratios ($P < 0.05$).

3.6 Water Holding Capacity (WHC)

This shows that all the substrates presented high water holding capacity values irrespective of the formulation employed (Fig. 4). The water holding capacity of compost samples of 60%, 70% and 80% was found to be high at 0.55 BWS ratio where high BSDS that has large porosity [6] to retain water. However, the 70% moisture content experiment contained higher WHC than other compost samples. In the 50% moisture content experiment, the microbial activity of degrading of organic compounds were slow

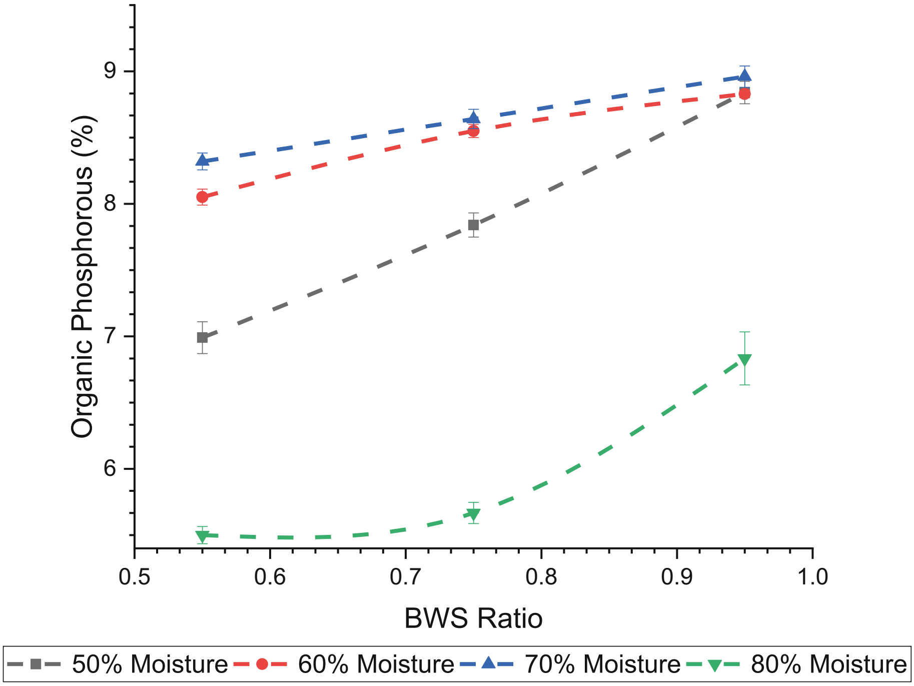

Fig. 3. Organic phosphorus percentage of final compost as function of BWS ratio

due to the lower moisture content. The results revealed that the water holding capacity of all compost samples (57–62%WHC) are greater than the control soil (22%WHC).

Organic matter of compost adds to the water retention capacity of a soil because humus particles absorb water and increase the ability of a soil to retain moisture against drainage due to the force of gravity [25].

3.7 Metal Analysis of Compost

The nutrient contents of compost samples were characterized and were found containing essential plant nutrients as seen in Table 4 and their value is lower than the maximum permissible limit. As it can be seen in Table 3, the heavy metal concentration in the final compost is found far below permissible limit. The brewery spent diatomaceous slugdes (BSDS) that was used in the compost samples was scavanging heavy metal ions and there was reduction of heavy metals as compared to raw sludges because BSDS contained high silicon dioxide content shows scavanging effect for metal ions [18]. The compost samples can be used as organic fertilizer [15].

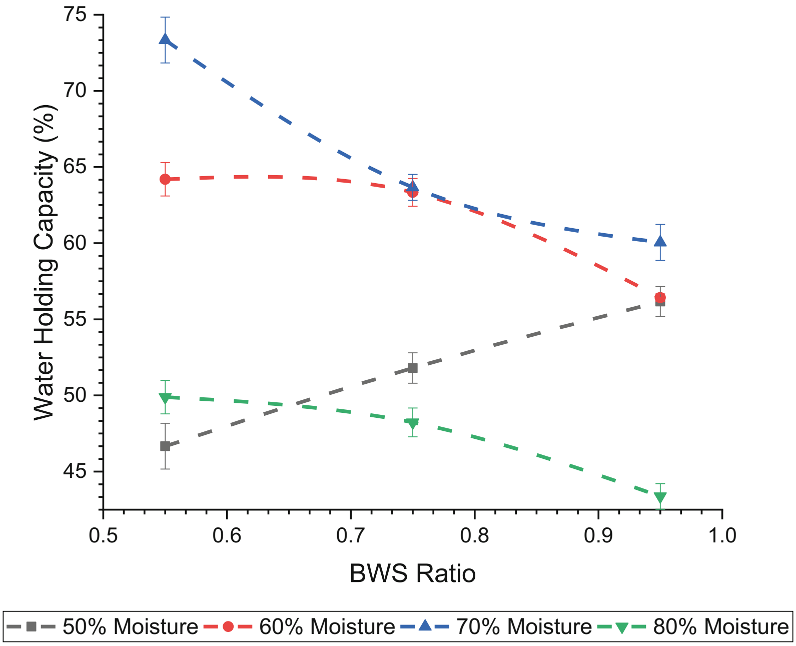

Fig. 4. Water holding capacity (%) of final compost as function of BWS ratio

Table 3. Metal composition of the final compost

Metals	Value	Limit [28, 29]
Copper, Cu (mg/kg)	5.36 ± 0.03	70
Sodium, Na (g/kg)	111.29 ± 0.91	214
Potassium, K (mg/kg)	123.67 ± 1.39	1608
Magnesium, Mg (mg/kg)	846 ± 82.58	1478
Calcium, Ca (mg/kg)	4004 ± 408.3	5789
Iron, Fe (mg/kg)	26.34 ± 0.48	266
Lead, Pb (mg/kg)	0.273 ± 0.005	45
Cadmium, Cd (mg/kg)	0.475 ± 0.004	0.7
Mercury, Hg (mg/kg)	0.165 ± 0.004	0.7

3.8 Microbiological Analysis of Composts

In present study, composting temperature was found higher than 55 °C [26]. The microbiological analyses results of the final composts are presented in Table 3. Compost samples were analysed for coliforms, salmonella and Escherichia coli (E. coli). The results indicate that composting practices is effective for pathogen reduction of brewery sludges composting. As it can be seen in the table, Salmonella, which is considered a

Table 4. Microbiological groups in composts and legislation

	Concentration	EPA standard
E.coli (CFU/g)	4.24 ± 0.11	$<$100 MPN/g
Salmonella (CFU/25 g)	Not detected	$<$1/25 g
Fecal coliform (CFU/g)	2.87 ± 0.4	$<$100 MPN/g

*MPN = Most Probable Number

good index for the hygienic status of the composts [27] was not detected in the composts. The compost samples contained total fecal coliforms (TFC) and E. coli lower than the permissible EPA standard value, which indicates that composting practice reduces the pathogen concentrations and hygienic status of the compost samples.

4 Conclusion

Brewery is among the industries known for production of byproducts (spent grains, spent yeast) and sludge (BWS and BSDS) from the brewery wastewater treatment plant at different stages of manufacturing process. Aerobic composting experiment was conducted for a wide range of sludge moisture and sludge ratio. Based on the findings of this study, it was found that the compost material obtained by aerobic degradation of the raw sludge is within the standard organic fertilizer requirement. This research revealed that brewery sludge compost is a rich source of nitrogen, phosphorous and potassium with other plant nutrients. Besides, analysis of selected heavy metals in brewery sludge composts suggested that Lead, Mercury and Cadmium were found below the threshold levels and the compost can be safely used. Water holding capacity of the compost products were found between 57 and 62% WHC, which is much higher than the soil (22% WHC). This suggested that, which is linked to high organic matter, high cation exchange capacity and other nutrients that can improve water holding capacity of soils. It can be used for sustainable agricultural land reuse. The future work is to study compost samples to soil ratio and effect of compost fertilizer on composition of vegetable.

Acknowledgements. The authors would like to thank Bahir Dar University, Bahir Dar Institute of Technology for financial supports. The authors honestly acknowledge laboratory technicians for their patience, friendly approach and collaboration for accomplishment of this work.

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