International Journal of Food Science and Agriculture, 2024, 8(1), 26-33
https://www. hillpublisher. com/journals/jsfa/
ISSN Online: 2578-3475
ISSN Print: 2578-3467

Remediation of Copper Contaminated Soil by Char Derived from Used Disposable Baby Diapers

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How to cite this paper: A. Kavinthini, A. D. Igalavithana. (2024). Remediation of Copper Contaminated Soil by Char Derived from Used Disposable Baby Diapers. *International Journal of Food Science and Agriculture*, 8(1), 26-33.
DOI: 10.26855/ijfsa.2024.03.004

Received: February 17, 2024
Accepted: March 14, 2024
Published: April 11, 2024

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Abstract

Plastic, including used disposable baby diapers (UDD), is a growing soil pollutant due to increasing single-use plastic consumption. UDDs contain 50% plastic such as super absorbent polymers that take over 100 years to degrade. This study focused on pyrolysis as a recycling method to produce plastic char from UDDs, evaluating its efficacy in the soil as an amendment to immobilize the copper in contaminated soil. Uncontaminated soil was spiked (240 mg/kg) with copper (ll) sulphate. Based on the thermogravimetric analysis of UDD, 550°C was selected as the pyrolysis temperature. The plastic char produced was characterized before being applied to the soil. The contaminated soil was incubated after applying plastic char at the rates of 0% (control), 1%, and 2.5%. Immobilization of Cu was measured after one month of incubation by single extraction using 1M NH4OAc and consecutive extraction using 0.01 M CaCl2 methods. The soil pH, electrical conductivity, and availability of Na, Mg, K, P, Ca, and N were analyzed. The consecutive extraction analysis revealed a significant ($p < 0.05$) reduction in extractable Cu concentration in the soil treated with plastic char and the immobilization percentage of copper by plastic char was 86%. The decrease was uniform for both plastic char doses. The properties of plastic char, such as its high pH (11.27), ash content (36.68%), and Electrical conductivity-EC (0.03 dS/m), likely contributed to increasing soil pH and facilitating Cu immobilization. Furthermore, the presence of functional groups such as C-H, C=O, OH, N-H, P-H, and C-Cl and carboxylic acid-like functional groups on the surface of plastic char, along with its high fixed carbon content (48.14%), likely supported the immobilization of Cu. In addition, it has 70 $cmol^{+}kg^{-1}$ at 550°C. These findings indicate that plastic char derived from UDD can be effectively utilized to immobilize Cu in contaminated soil. The pyrolysis of UDD represents a promising waste management practice that minimizes environmental pollution.

Keywords

Soil amendment, Disposable baby diapers, Immobilization, Plastic char

1. Introduction

The environmental accumulation of plastic items such as plastic bottles, polythene bags, and others is commonly referred to as plastic waste or plastic pollution. This accumulation poses a threat to the survival of plant and animal habitats as well as their health. Plastic recycling is becoming increasingly crucial as plastic production continues to escalate worldwide. According to [1], plastics rank among the most widely used polymers. In today's world, agricultural, urban, and modern activities collectively contribute to significant waste production, exacerbating environmental challenges. In 2009, approximately 140 billion disposable diapers were produced globally. Particularly, the rapid increase in the usage of adult disposable diapers by elderly individuals or those requiring nursing care has led to a surge in diaper production in Japan, from 7.9 billion in 1999 to 14.5 billion in 2011. One prominent type of plastic waste is disposable baby diapers (UDD). While single-use baby diapers offer high convenience for infant care, their disposable nature poses significant challenges to waste management [2]. Diapers typically consist of outer, middle, and inner layers, each comprising distinct materials. The acquisition and distribution layer (ADL) contains fluff pulp made from protein-based fibers like silk and wool, cellulose fibers like cotton and wood pulp, and super absorbent polymers (SAPs). The outer layer comprises polypropylene (PP) and polyethylene (PE), while the middle layer contains absorbent cores made from cellulose- and protein-based fibers, with PP and PE in the inner layer [2]. The disposal of used diapers poses a significant challenge, with annual production projected to reach nearly US$ 71 billion by 2022. Various technologies, including composting, thermal pyrolysis, anaerobic digestion, dark fermentation, and incineration, offer potential for plastic recycling globally [3]. Effective recycling methods for used diapers include biodegradation and thermal pyrolysis. The composition of used baby diapers, which includes biomass (cellulose), polymers (polyethylene and polypropylene), and adsorbent materials, complicates conventional disposal and valorization strategies such as landfilling and trans-esterification. Pyrolysis offers flexibility in reallocating carbon compounds from biomass and waste materials to produce three types of pyrolysis products (liquid oil, gases, and char) in an oxygen-free environment. Trace element contamination is a well-known global environmental issue resulting from natural and human activities, polluting both water and soil. Trace minerals, being non-biodegradable, persist for extended periods. Traditional methods utilize organic and inorganic amendments to address this issue. However, recent trends involve using plastic char as a remediation tool to immobilize soil trace minerals due to its properties. Plastic-char, with its numerous pores, large specific surface area, high cation exchange capacity (CEC), and abundant surface functional groups, exhibits excellent absorption of trace minerals. This research aims to understand environmental issues and propose solutions by examining user attitudes and waste disposal practices. It investigates the potential of plastic-char derived from used baby diapers as a remediation tool for intentionally contaminated soil samples with trace elements (Cu). The study compares results from an incubation experiment using alluvial soil with findings from previous research. If the hypothesis holds, subsequent trials can determine the appropriateness of applying plastic-char to contaminated soil, potentially offering an eco-friendly and effective remediation method in the near future [4].

2. Methods

2.1 Soil sampling and characterization

The representative soil sample was obtained from 7° 15' 51.77" N, 80° 35' 39.88" E in soil of Kandy series at benchmark (KN2). Collected soil was air dried and sieved into < 2.00 mm and then the desired copper concentration of soil that is needed for remediation measures was found 240.00 mg for one kg of soil was purposefully spiked by using copper sulfate salt. Following this, the contaminated soil was incubated for a week, after which the soils were analyzed to determine their physicochemical properties. Soil texture was determined by the pipette method as described [5]. Soil organic carbon percentages were calculated using the loss on ignition method by [6]. A 1:5 soil: distilled water suspension was used to determine soil pH and electrical conductivity (EC) [7].

2.2 Preparation of char derived from used disposable baby diapers (UDD)

The process of converting used disposable baby diapers (UDD) that contain only liquid waste (human excreta) into plastic char involves several steps. Firstly, the UDD was cut into small pieces and left to dry in the air for a couple of weeks. Then, the dried UDD was placed in a stainless-steel reactor inside a muffle furnace (Hobersal, Spain). The container was covered with a tightly fitting lid to minimize air exposure during the pyrolytic process. The UDD was subjected to pyrolysis at a temperature of 550°C for 30 minutes. Once the process was complete, the resulting product was crushed and sieved into particles smaller than 2.00 mm. These particles were stored in an airtight container until they were ready to be used. Hereafter, the prepared product was denoted by UDD_{550}, where 550 indicates the pyrolytic temperature [8].

2.3 Characterization of char made from used disposable baby diapers (UDD550)

Physical and chemical analyses were conducted to provide the basic properties of UDD_{550}. The yield of the UDD sample was measured before and after heating at 550°C for 30 minutes where the yield was defined as the percentage of plastic char weight after the pyrolytic process compared with the initial weight of the feedstock (41.30%). The percentage of moisture, volatile matter, ash, and fixed carbon were determined. The pH and electrical conductivity were measured by 1:20 (sample/distilled water) ratio method using a pH meter (EUTECH pH 510) and EC meter (EUTECH CON2700), respectively. The content of total nitrogen was measured by using Kjeldahl method. Available phosphorus was extracted by the Olsen P method. The cation exchange capacity was measured by using semi-micro Schollbenger method with 1M ammonium acetate solution at pH 7. Heavy metal and exchangeable cations were measured by atomic absorption spectrophotometer (Agilent Technologies, Malaysia). The FT-IR spectra were collected with a spectrometer using KBr pellets with a wavenumber ranging from 500 to 4000 cm^{-1} [9].

2.4 Soil incubation study and soil analyses

An incubation study was conducted to evaluate the potentials of UDD550 and Copper (Cu) immobilization in soil. Copper-contaminated soil (200 g) and 1%, 2.5% (w/w) of an amendment UDD_{550} were mixed thoroughly and placed into a 400 ml high-density polyethylene bottle. The lid of the container was fixed and holes were riled on the top of the lid creating the aerobic conditions during incubation. The water content of the soil was adjusted to 70% of water water-holding capacity and incubated at 25°C in the dark for 29 days. Four replicates were maintained for each treatment. Once the incubation was completed soil was air dried and the soil chemical properties were determined. Single and consecutive extractions determined the availability and mobility of the heavy metal in soil. Single extraction of metal was performed with 1M NH_4OAc at pH 7 solution at a ratio of soil: NH_4OAc of 1:5 metal mobility kinetics were evaluated by consecutive extraction of soils using a 0.01M calcium chloride ($CaCl_2$), pH 5.5 solution. The soils were extracted consecutively four times by shaking a 1:5 suspension of soil: $CaCl_2$ for 24 hours at 25°C [10].

2.5 Statistical analysis

Data were tested for normal distribution [11] and as all the data were normally distributed, they were further analyzed with parametric tests to evaluate the effects of amendments. Pearson correlation analysis was used to explore relations between extractable element contents and the soil properties in the treatments. One-way analysis of variance (ANOVA) followed by Tukey's honestly significant difference (HSD) test at $p < 0.05$ was used to compare the treatments and identify significant differences [10].

3. Results

Prior to incubation analysis with plastic char initial soil properties were analyzed and resulted as, based on the USDA soil textural triangle, the texture of the taken soil was sandy loam (clay -2.27 %, silt-7.84 %, sand-82.72 %), and the soil order was Ultisol. The organic matter content in taken soil was 1.19%, which had a low amount of organic matter content. The moisture content of the air-dried soil was 2.11% and the water-holding capacity of the soil was 21.25 %.

The chemical properties of the plastic-char were determined and shown in below Table 1. As a result, the pH of plastic char was alkaline. The electrical conductivity (EC) of the plastic char sample was high. Total nitrogen content was low in amount. After digesting the ash component of plastic char available phosphorus (P) content and also available potassium (K) content in the plastic char were analyzed. It was significantly high in amount. Inherent copper concentration was observed, it was under permissible limits by FAO.

Table 1. The properties of plastic char were measured by using the standard method. UDD_{550} was compared with three different temperatures yielded plastic char according to the data obtained from Oh and Shinogi (2013).

UDD samples	pH 1:20	EC dSm^{-1}	Total-N	Available P_2O_5 (g kg^{-1})	CEC $cmol_c$ kg^{-1}	Ash content %	Fixed carbon content %	Volatile matter content %	Moisture content %	Available potassium content %
UDD_{500}	11.31	0.15	0.39	4.34	42.03	19.10	20.11	60.22	0.57	3.42
UDD_{550}	**11.27**	**0.33**	**0.39**	**3.78**	**70.00**	**48.84**	**36.68**	**13.95**	**0.53**	**6.26**
UDD_{700}	12.03	0.44	0.12	4.34	87.06	51.65	42.44	5.40	0.51	2.95
UDD_{900}	12.62	1.22	0.03	5.79	95.09	52.18	45.08	2.33	0.40	9.90

FT-IR was used to identify the specified chemical functional groups present in the UDD sample. FT-IR emission spectra of the UDD_{550} are shown in Figure 1. In the spectra of UDD_{550}, various bands are observed for OH (3750 cm^{-1}), C-H (2750-3500 cm^{-1}), C=O (1630 cm^{-1}), N-H (1555 cm^{-1}), Carboxylic acid (2250-2500 cm^{-1}), P-H (1057 cm^{-1}) and C-Cl (713 cm^{-1}). The intensity of the broad and strong peak around 2250-2500 cm^{-1} which contained a high amount of carboxylic group.

Single extraction analysis was performed to find the treatment effect on incubated soil copper concentration by using 1 M NH_4OAc. It resulted as Figure 2, provided the mean values of the copper concentration of the treatments B1 and B2.5 were lower than the control (B0). There was a significant difference between the control and treatments B1 and B2.5. However, there was no significant difference between treatments B1 and B2.5, indicating that the application rate of plastic char (1%, 2.5%) did not significantly affect the immobilization of copper-contaminated soil.

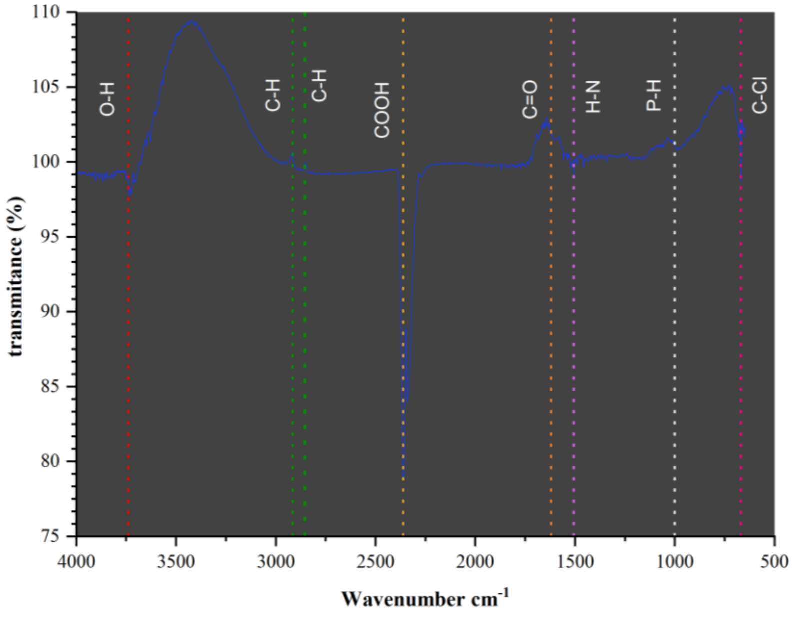

Figure 1. FT-IR emission spectroscopy illustration of plastic char. Dotted line bands indicate the functional groups on the surface of the plastic char at 550°C. Colored dash lines indicate the wave number where the emission happened. Each and every peak showed the presence of functional groups.

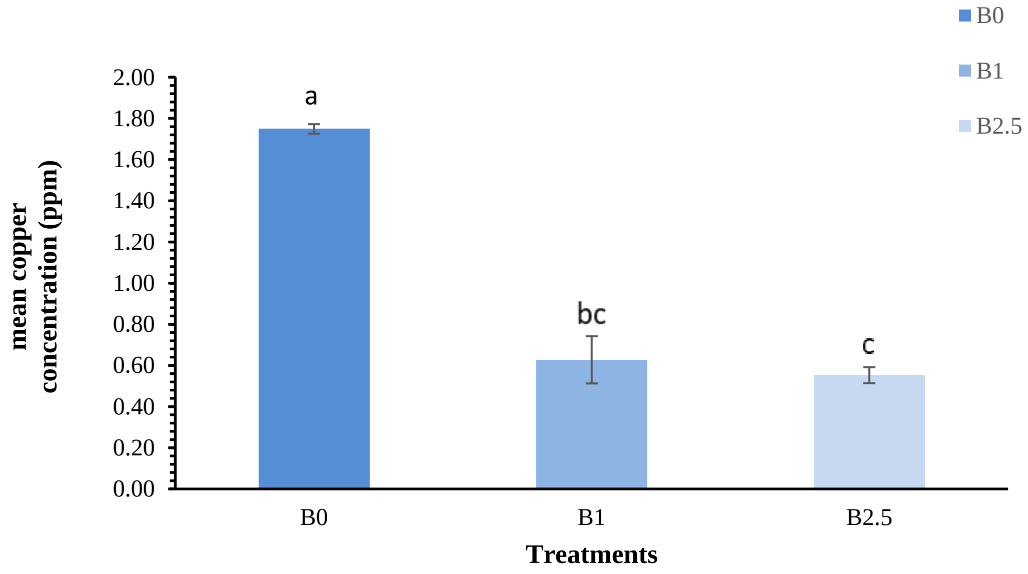

Figure 2. 1 M NH4OAc extractable Cu in incubated soil. vertical lines indicate the standard deviation. The letters above the vertical bars indicate statistically significant differences at p<0.05 (Tukey's post hoc test). Treatments: BO denotes control, B1 - 1 % w/w of plastic char, and B2.5 – 2.5 % w/w of plastic char.

Consecutive analysis was performed by using 0.01M $CaCl_2$ at pH 5.5 to find the kinetics of heavy metal release. The addition of plastic char as a soil amendment significantly reduced the mobility of the metal, particularly copper, in all four extractions. Compared to the control, both treatments (B1 and B2.5) showed a significant reduction in the concentration of available copper in the first and second extractions. Subsequently, in the third and fourth extractions, copper concentration was below detection limits due to the plastic char treatment. There was a significant difference between the control(B0) and both treatments.

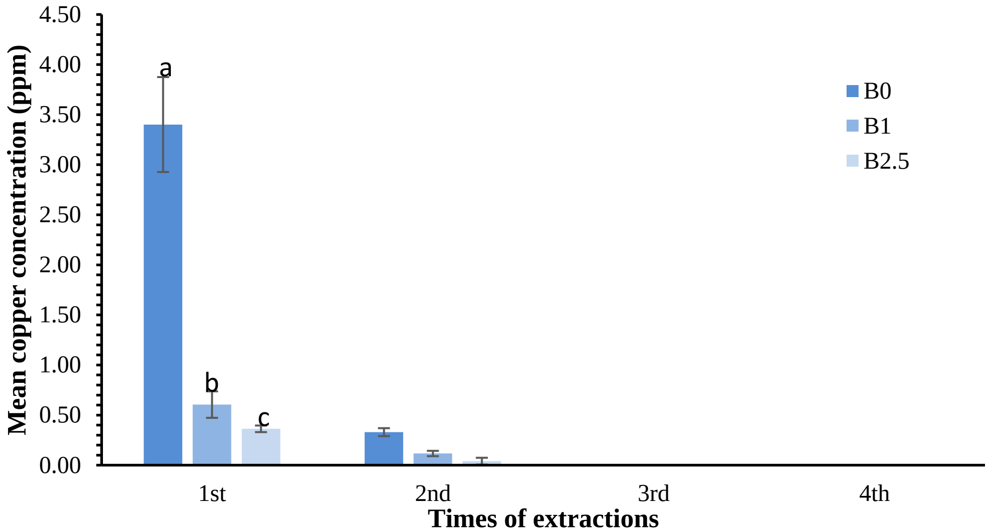

Figure 3. Consecutive extraction of copper in incubated soil by 0.01M CaCl2, vertical lines indicate the standard deviation of the letters above those statistically significant differences at p<0.05 (Tukey's post hoc test). Treatments: B0- control, B1- 1% w/w of plastic char and B2.5- 2.5% w/w of plastic char.

The application of plastic char in the incubated soil increased the cation exchange capacity (CEC) as illustrated by which was dependent on the application rate. The trend observed was as follows: control<treatment 1<treatment 2.

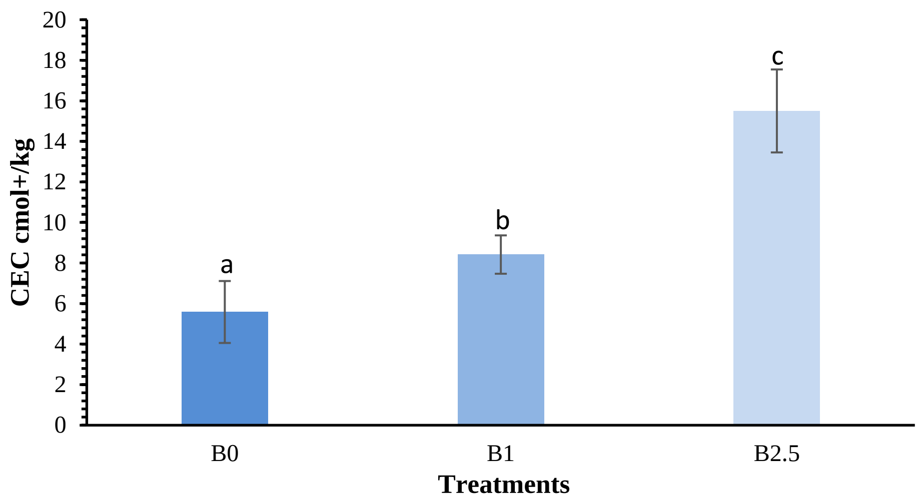

Figure 4. The cation exchange capacity of incubated soil. Vertical lines indicate the standard deviation. The letters above the vertical bars indicate statistically significant differences at p<0.05 (Tukey's post hoc test). Treatments: BO indicates control, B1 - 1 % w/w of plastic char, and B2.5 – 2.5% w/w of plastic char.

The organic matter content of the initial soil (1.19%) was increased with respect to the application rate of plastic char. Based on organic matter content was increased up to 1.31%.

After twenty-nine days of incubation, concentration of copper and soil chemical and physical parameters were observed. Based on the correlation results, the pH of the incubated soil was found to be negatively correlated with a reducing copper concentration in single extraction. The pH increases leading to the immobilization of copper, with a high negative correlation observed (r= -0.969) between pH and copper concentration. The electrical conductivity (EC) of the soil was also measured and found to be positively correlated (r= -0.809) with copper immobilization in a single extraction. Sodium (Na) concentration was measured and found to be increased with respect to decreasing copper concentration. Sodium concentration showed a negative medium level of correlation (r= -0.781) with copper ion concentration. Magnesium (Mg) concentration was shown positive correlation with reducing copper concentration (r= +0.937). Available phosphorus (p) concentration was observed negatively correlated (r=-0.887) with decreasing copper concentration. Potassium (K) concentration showed an increasing (r= -0.714) trend as well as calcium (Ca) concentration decreased with reducing copper concentration (r= +0.915) respectively with reducing copper concentration. Available nitrogen (N) showed decreasing trend (r= +0.787) with reducing copper concentration.

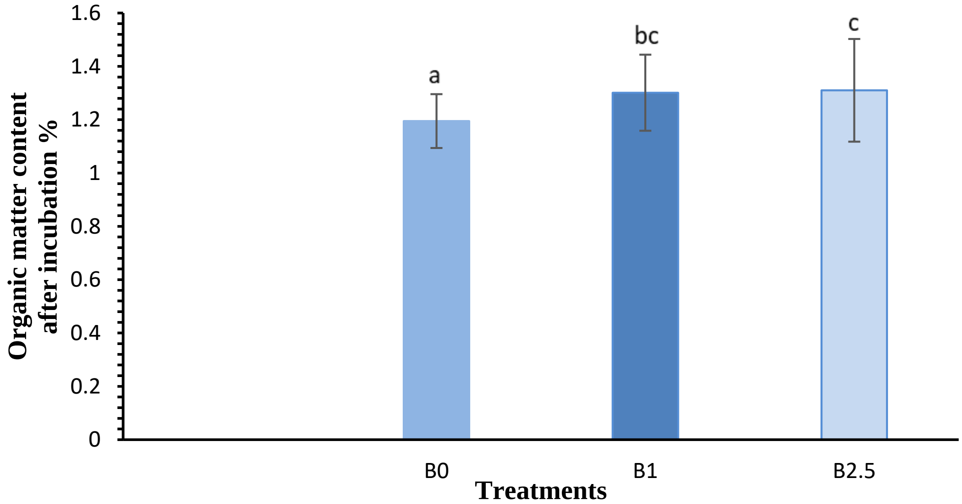

Figure 5. improvement of organic matter content in copper contaminated soil was measured after incubation period. Vertical lines indicate the standard deviation. The letters above the vertical bars indicate statistically significant differences at p<0.05 (Tukey's post hoc test). Treatments: BO indicates control, B1 - 1 % w/w of plastic char, and B2.5 – 2.5% w/w of plastic char.

Table 2. Chemical properties of the soil after incubation. Data are presented as the mean with standard deviation. The letters following the parenthesis indicate a significant difference at p<0.05 (Tukey's post hoc test)

Treatment	pH	EC	Na mg/kg	K mg/kg	Mg mg/kg	Ca g/kg	N g/kg	P mg/kg
B0	7.50±0.05 a[1]	0.07±0.00 a	0.23±0.03 a	11.63±0.43 a	2.35±0.05 a	0.70±0.26 a	0.51±0.12 a	0.16±0.05 a
B1	9.50±0.07 b	0.71±0.04 b[2]	11.45±8.60 b	31.72±3.12 b	1.72±0.31 b	0.13±0.05 b	0.37±0.03 b	0.89±0.18 b
B2.5	10.34±0.03 c	1.78±0.04 c	20.6±6.35 c	98.86±6.40 c	1.56±0.11 c	0.10±0.02 c	0.20±0.03 c	1.51±0.03 c[3]

4. Discussion

During the pyrolytic thermal decomposition of used diapers analyzed by thermo gravimetric analyzer, the main weight reduction phase occurred between 30.05°C and 200°C. The weight of the diapers decreased from 100.56% to 81.92% due to dehydration, removing up to 18.64% of their moisture content. Typically, this dehydration and water removal stage occurred during the initial pyrolysis of lignocellulose materials in used disposable baby diapers. Figure 6 shows the primary decomposition of UDDs during the temperature range of 400 to 600°C. This process led to a significant disintegration of the diapers, resulting in a substantial weight reduction of up to 30.96%. It is highly probable that the decomposition of the diapers, composed of hydrocarbons, polymers, and lignocellulose components (cellulose, hemicellulose, and lignin), caused them to transform into volatile matter. Between temperatures of 800 to 1000°C, additional decomposition and reduction in weight were observed, indicating the occurrence of passive pyrolysis. This stage involved a gradual and stable breakdown of the fixed carbon present in the UDDs. Upon analysis, it was found that these diapers contained 48.14 weight % fixed carbon, which could be effectively recovered through pyrolysis to generate valuable carbonaceous materials, such as solid plastic char [12]. As a result of characterizing the properties of plastic char (UDD_{550}), the pH of plastic char was alkaline. This extreme pH value was observed due to basic cationic salts retained in the ash component of the plastic char samples after the pyrolytic process. According to [9], adding pyrolytic solid products to acid soils, pH increases, because there is an increased concentration of alkaline metals such as K^+, Ca^{2+}, and Mg^{2+}. The high pH is a useful attribute of this plastic char for neutralizing soil acidity when added to acidic soil; thus, it could be used to remediate acidic soil from heavy metals. The high electrical conductivity (EC) of the plastic char sample was observed due to the

[1] a is the parenthesis
[2] b is the parenthesis
[3] c is the parenthesis, which indicates all three treatments are significantly different in above-mentioned parameters.

release of different mineral salts, which refer to the presence of dissolved ions in the plastic char. This value indicated the plastic char would increase the soil EC at a significant level when used as a soil amendment and the EC value showed a positive correlation with pyrolytic temperature according to [9]. Although UDDs contained lots of nitrogen content because of urine due to high pyrolytic temperature (550°C) volatilization of ammonia would happen. Therefore, a low amount of total nitrogen content was observed in plastic char. The total N content reduces with increasing pyrolytic temperature based on [9]. After digesting the ash component of plastic char available phosphorus (P) content and also available potassium (K) content in the plastic char were analyzed. This was because urine contained large amounts of salts. Inherent copper concentration was negligible. For the incubation experiment, the soil was spiked with copper sulfate salt. A concentration of 240 mg/kg soil was chosen because the maximum permissible level of copper in the soil, according to FAO, is typically between 50-100 mg/kg. Additionally, at a concentration of 240 mg/kg, seedling emergence will be affected. To understand the kinetics of heavy metal after incubation, a single extraction method was performed and it resulted in the ability of plastic char to immobilize copper due to its high specific surface area and functional groups on the surface. Copper can also be immobilized by microorganisms, clay minerals, inherent organic matter, Mn and Fe oxides, and carbonates-bound precipitation. In the control, copper immobilization occurred because of bound precipitation [10], and consecutive extractions were done using 0.01M $CaCl_2$ at pH 5.5, mimicking the conditions in the rhizosphere. As a result both plastic char rates effectively reduced the mobility of soil copper compared to the control under acidic pH conditions as well [10]. The plastic char used in this study contains oxygen-containing functional groups, such as carboxyl and carbonyl, which can enhance the CEC of the soil (Figure 1). This is because of the carboxyl and carbonyl, which can enhance the CEC of the soil. This is because the carbonyl groups deprotonate readily in water, leading to a more negative charge on the plastic char surface. However, it is important to note that the effect of plastic char on CEC can vary depending on the type of feedstock used and the pyrolytic temperature applied. The CEC of UDD at 550°C was 70 $cmol^{+}kg^{-1}$ to compare with 500, and 700 °C [9]. After incubation, the concentration of copper and soil chemical and physical parameters were observed. Based on the analysis pH and EC of plastic char are positively correlated with reducing copper concentration. Because of its high ash content (Table 1), it contains basic ions which was the reason for increasing pH and EC. Nitrogen showed a negative correlation with reducing copper concentration. Increasing pH inside the container leads to ammonia volatilization and is positively correlated with reducing copper concentration. Na, P, K showed a negative correlation with reducing copper concentration as a result of high ash content because babies' urine contains more sodium and potassium phosphate and it contains less amount of Ca and Mg salts. Therefore, calcium and magnesium showed a negative correlation with reducing copper concentration [13].

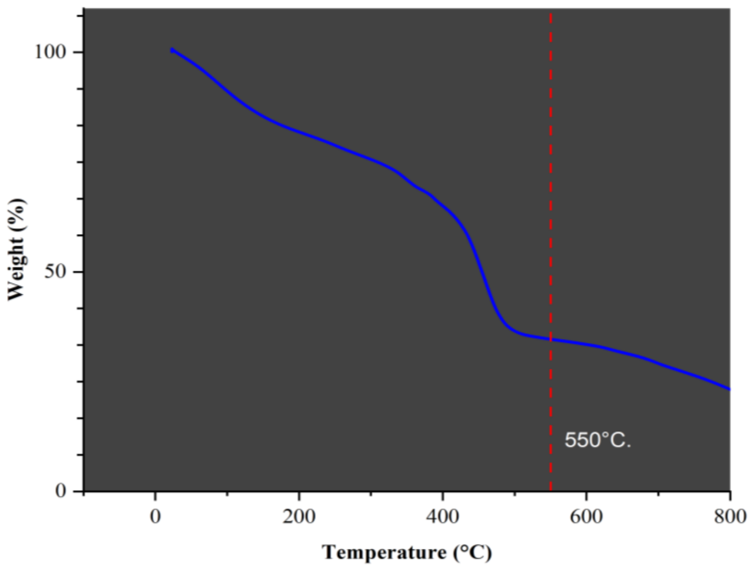

Figure 6. Thermo gravimetric analysis of used disposable baby diapers (UDD) which is used to find the suitable temperature for pyrolysis of UDD. Reference dash line indicated the temperature where the weight % is not varied suddenly. This graph depicts how the weight percentage reduces with increasing temperature.

5. Conclusion

Disposable baby diapers (UDD) are becoming a significant concern in many nations. Experimental results demonstrate that the pyrolytic process efficiently reduces their volume. Characterization of the resulting plastic char reveals high fixed carbon and ash content, low volatile matter content, and high pH, EC, and CEC. This suggests that plastic char can be reused as a soil amendment by increasing pH and releasing nutrients due to its high ash content. It contains negligible amounts of inherent copper (Cu) concentration, posing no harm to the environment. The large surface area and numerous functional groups (such as C-H, C=O, OH, N-H, P-H, C-Cl, and carboxylic acid) on plastic char facilitate the immobilization of copper in treated soil. Consecutive extraction analysis demonstrates a significant reduction in copper concentration in soil treated with plastic char compared to the control, although there was no significant difference between the two application rates of plastic char. These findings indicate that plastic char derived from UDD can effectively immobilize copper in contaminated soil ($p < 0.05$) and improve soil pH. In conclusion, pyrolyzed UDD represents an efficient waste management option without polluting the environment.

Acknowledgements

This paper was supported by University Research Grants and all the research facilities were provided by the Department of Soil Science, Faculty of Agriculture, University of Peradeniya, Sri Lanka.

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