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RESEARCH

Changes in Total and Available K Contents in Feldspar and Mica Co-pyrolyzed with Poultry Litter at Different Temperatures

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ABSTRACT

Plant available potassium (K) content is very low in K-bearing mineral ores present in Sri Lanka and that restrict their use in crop production as K fertilizer sources. Heating of minerals can increase their plant available forms of nutrients. The objective of this experiment was to study the effect of direct heating of K-bearing minerals at different temperatures and co-pyrolyis with poultry litter (PL) at different rates on their total and available K contents. Poultry litter was co-pyrolyzed with 0, 1 and 5% (w/w) of feldspar and mica at 350, 500, 650 and 900 °C. K-bearing minerals were also directly heated at these temperatures. With direct heating the mineral, total K contents of feldspar (6.2%) and mica (4.5%) were significantly ($p<0.001$) increased by 10% and 18%, respectively at 500 °C and then decreased by 33 and 65%, respectively at 900 °C. Available K contents in feldspar (0.05%) and in mica (0.03%) either decreased significantly (feldspar) or did not change (mica) upon direct heating. Increasing pyrolysis temperature up to 650 °C increased both total (4.2 to 6.4%), and available K (3.2 to 6.1%) contents in poultry litter biochar (PLB). Co-pyrolysis of PL with feldspar or mica at 1 or 5% rates did not improve the total or available K contents in PLB at these temperatures. We concluded that heat treatment of locally available feldspar and mica or co-pyrolyzing them with PL are not effective technologies to increase their utilization in agriculture.

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INTRODUCTION

Potassic fertilizers are essential for obtaining maximum yields in most crop production systems. Majority of the agricultural soils of Sri Lanka are deficient in plant available potassium (K) due to inherent characteristics in parent materials and leaching due to heavy rains and low cation exchange capacities (Kumaragamage and Indraratne, 2011). Presently, farmers are struggling in managing soil fertility due to exorbitant prices of K fertilizers in the market and the removal of material subsidy of fertilizers. As a result, supplementing K deficiency in agricultural soils with imported fertilizers is almost cost prohibitive. Therefore, it is imperative to investigate alternative indigenous K resources for the production of K fertilizers. It is more economically viable to transform fixed slow-release sources of K such as K-bearing minerals into plant available K forms (Hu et al., 2021).

Feldspar and mica are primary alumino-silicate minerals and found abundantly in mineral deposits scattered throughout Sri Lanka. Feldspar can be found in Rattota, Namaloya, Koslanda, and Balangoda localities (Rajapakse, 2022) and mica can be found in Kaikawala, Medawachchiya, Rathnapura, Matale and Dambulla (Herath, 1975). Currently, these minerals are being mined for ceramic and glass industries (Rajapakse, 2022) but have a potential to be used as a substitute for imported K fertilizers in Sri Lanka (Nivethithan et al., 2022).

Although total K contents in feldspar (14.03%) and mica (9.03%) are considerably high (Manning, 2010), availability of that K to plant is very low. Bakken et al., (1997) observed that only 0.24% to 0.49% of K in feldspar was soluble in 7N HNO3 solution. Basak and Biswas (2009) observed only 0.02% water soluble K in waste mica samples obtained from a mica mining site in India. According to Bakken et al. (1997) the estimated K-uptake in soils treated with K-feldspar was very low as K bound in the mineral K-feldspar is nearly unavailable for plants. Therefore, the value of alumino-silicate K-bearing minerals as plant nutrient sources is negligible. Increasing K availability in these minerals is a pre-requisite to heap maximum benefits from them as a fertilizer.

Several studies have been conducted to increase K availability in K-bearing minerals. Application of K-bearing minerals with K solubilizing bacteria (Liu et al., 2006; Zarjani et al., 2013; Meena et al., 2016), treating with an acid before soil application (Lim et al., 2003; Wafaa et al., 2015) are some of the technologies that have been used with variable success rates. Another approach to promote modification in mineral structure is by subjecting them to thermal treatment. Thermal treatment at different temperatures between 300-1200 ℃ has increased K^+ and Na^+ availability in phonolite rock powder due to changes in mineral structures (Teixeira et al., 2015). Typically, when K-bearing minerals are heated above 800 ℃ the release of K^+ from the mineral matrix can be promoted, making them more available for plant uptake (Francisco et al., 2007). They have also reported that heat treatment can lead to the fragmentation and pulverization of the K-mineral particles while resultant smaller particles increase the surface area available for interactions with the soil solution, enhancing K dissolution and subsequent uptake by plants.

Poultry litter (PL) is a common soil amendment used in Sri Lanka. It is a mixture of poultry manure and bedding materials mainly comprised of rice husk in countries where rice is being cultivated as a main crop (Chan et al., 2008). Rice husk is also a good source of K (Song and Guo, 2012). Poultry litter contains about 41.77 ± 1.37 g/kg of K (Suthamathy and Seran, 2013). However, there are some environmental concerns when raw poultry litter is applied to agricultural soils. Poultry litter is a source of antibiotic resistance determinants to the environment (Herath et al., 2016) and may contain pathogenic organisms like Salmonella, Campylobacter and generic Escherichia coli (Pope and Cherry, 2000). These negative effects on the environment could be negated by converting them into biochar through pyrolysis (Muhammad et al., 2020). Converting PL into biochar through pyrolysis and their land application has gained popularity as a sustainable soil fertility management strategy in many agricultural production systems (Masocha and Dikinya, 2022). On the other hand, Song and Guo (2012) revealed that as pyrolysis temperature increased incrementally from 300 to 600 °C, it has increased total K content from 69.28 in PL to 91.51 mg/kg in poultry litter biochar (PLB) and available K contents from 32.01 in PL to 44.61 mg/kg in PLB.

Pyrolysis is an exothermic reaction and heat within the pyrolysis reactor could be used to increase the plant available nutrient contents in minerals. For example, co-pyrolysis of rice husk with rock phosphate has increased the phosphorous availability in rice husk biochar (RHB) at temperatures ranging from 350 to 600 °C (Dissanayake et al., 2018). Co-pyrolysis of waste maize stover with a low solubility igneous rock phosphate has significantly increased the amount of P as measured by citric acid extraction (Tumbure et al., 2020). Basak et al. (2021) also have revealed that P availability in biochar mineral complex could be increased by the hydrothermal reaction of biochar with rock phosphate. However, published research is limited regarding the use of alternative low-cost K-bearing minerals in co-pyrolytic process and their effect on K availability of the resultant biochar. We hypothesized that co-pyrolyzing K containing minerals with PL could increase the K availability in PLB. Therefore, the main objectives of this study were to determine the effect of direct heating at different temperatures on the total K and available K contents of feldspar and mica, and determine the effect of co-pyrolysis of PL with these K-bearing minerals at different rates and different temperatures on K availability.

METHODOLOGY

Pre-processing of feedstock

As feedstock materials poultry litter, two locally available K-bearing minerals, feldspar and mica, were used. Biochars were produced from the broiler litter collected from a farm of National Livestock and Development Board (NLDB), Karandagolla, Sri Lanka. K-bearing mineral samples were procured from the Department of Geological Survey and Mines Bureau in Sri Lanka. Mineral samples were crushed and ground in a laboratory ball mill, sieved through <0.5 mm sieve and stored in air-tight plastic containers until further analysis. Raw poultry litter was sundried for 3-6 days and sieved using a 0.5 mm sieve and the material remained on the sieve (particles >0.5mm) was used for pyrolysis (Dissanayake et al., 2018).

Production of co-pyrolyzed biochar

Poultry litter was co-pyrolyzed with the two K-minerals incorporated at three different rates (0, 1, and 5% w/w) and at four different temperatures (350, 500, 650 and 900 °C) in a muffle furnace using closed metal containers with a heating rate of 10 °C/min and residence time of 2 h. Poultry litter pyrolyzed at different temperatures without mixing with K-minerals were abbreviated as PLB_{350}, PLB_{500}, PLB_{650} and PLB_{900}. Poultry litter co-pyrolyzed with 1% feldspar were abbreviated as PLB-IF_{350}, PLB-$1F_{500}$, PLB-$1F_{650}$ and PLB-$1F_{900}$ while PL co-pyrolyzed with 1% mica were abbreviated as PLB-$1M_{350}$, PLB-$1M_{500}$, PLB-$1MBC_{650}$ and PLB-$1M_{900}$. Similarly, PL co-pyrolyzed with 5% K-minerals were also abbreviated (PLB-$5F_{350}$, PLB-$5F_{500}$, PLB-$5F_{650}$ and PLB-$5F_{900}$ and PLB-$5M_{350}$, PLB-$5M_{500}$, PLB-$5M_{650}$ and PLB-$5M_{900}$). The two K-bearing minerals were also heated to the same temperatures without mixing them with PL. Three batches of PLB, with or without K-minerals, were subjected to the above-described heat treatments. Each sample of biochar were ground with mortar and pestle and sieved to achieve a particle size range of 149–850 µm prior to analyses (Enders et al., 2012).

Characterization of biochar and K-bearing minerals

Triplicate samples were drawn from each biochar treatment and characterized for pH, EC, total K and available K contents. The pH and electrical conductivity (EC) were measured according to Singh et al. (2017) in a mixture of biochar and distilled water at 1:20 ratio. The sample was shaken for 1.5 h in an orbital shaker and the pH was measured using EUTECH pH 510 pH meter and EC was measured using EUTECH CON 2700 conductivity meter. Available K was extracted as described by Enders et al. (2012) and K content in the extracts was measured using flame photometer (JENWAY PFP7). Total K content in K-minerals and co-pyrolyzed biochar was determined after digesting them with aqua regia: HF (5:2 v/v) Sun et al., (2001) using ETHOS EASY MAXI-44 eT Advanced Microwave digester and determining the extracted total K content using the flame photometry (JENWAY PEP7).

Co-pyrolyzed biochar samples that have improved K availability were characterized for their structural properties. Selected samples were imaged after gold coating at 50 kV electron beam using a fully automated Scanning Electron Microscope (VEGA3 SEM, TESCAN) for SEM analysis. Fourier Transformed Infrared (FTIR) spectroscopy analysis was performed on finely ground (<0.05mm) biochar samples using the diffuse reflectance accessory in a Nicolet (IS50) IR spectrophotometer and compared with that of raw PL, K-minerals and relevant PLB.

Data processing and statistics

Simple one-way ANOVA was conducted to analyze the effect of temperature on total and available K contents, pH and EC of the PLB at different pyrolyzing temperatures and to analyze the effect of temperature on total and available K contents of the K-bearing minerals. A three-way ANOVA was conducted to analyze the interaction of temperature, rate and type of K-bearing minerals on K contents. When the 3-way interaction was found to be significant ($p<0.05$) a two-way ANOVA was again conducted to analyze the interaction effect between temperature and rate on K contents for each mineral separately. Significant interactions were further analyzed using DMRT mean separation technique using SPSS 16.0 version.

RESULTS AND DISCUSSION

The temperature effect was significant ($p<0.001$) for total and available K contents in PLB and K-bearing minerals. The pH and EC of PLB also varied significantly with temperature. The 3-way interaction among temperature, mineral type and the K-bearing mineral rates was significant ($p<0.05$) on the availability of K in co-pyrolyzed PLB samples but not on the total K contents.

Effect of heating temperature on quality of poultry litter biochar

Pyrolysis temperature had a significant ($p<0.001$) impact on properties of PLB. The pH increased from slightly alkaline (pH= 7.5) to alkaline (pH=10.41) with the increasing pyrolysis temperature from 350 to 650 ℃ (Figure 1a), which are similar to the reported values of poultry litter biochars produced at high temperature (Revell et al., 2012; Song and Guo, 2012; Wang et al., 2015). Increasing pyrolysis temperature enhance the ash content by removing acidic functional groups, which cause biochar to be more basic (Ahmad et al., 2012; Steiner et al., 2016). The high pH could be attributed to the major ions in the ash component in PLBs, i.e. K^+, Na^+, Ca^{2+}, Mg^{2+}, HCO_3^-, and CO_3^{2-} (Song and Guo, 2012).

The EC of PLB was in the ranged from 0.012 to 0.074 S/m. Raw PL had a significantly ($p<0.001$) high EC values compared to that of all biochar samples (Figure 1b). Guo et al. (2009) observed EC values in PLB could range from 0.1 to 2.17 S /m but lower than that of raw PL (5.02 S/m). The significant amount of urine and manure which are rich in salts containing $NH4^+$ and K^+ ions could have contributed to the high EC of raw PL. Considering its high salinity, raw PL should be applied to soil at controlled rates to avoid potential toxicity on crop seeds and seedlings. Therefore, converting raw PL into PLB before its use is a better practice in order to reduce the risk of salinity development in agricultural soils.

Potassium availability refers to the proportion of K that is accessible or usable by plants. In raw PL, K availability as a percentage of total K content (*i.e.* available K percentage) was 76.2%. The conversion of raw PL into biochar had a significant impact on both total and available K contents. Total and available K contents increased with increasing pyrolysis temperature from 350 to

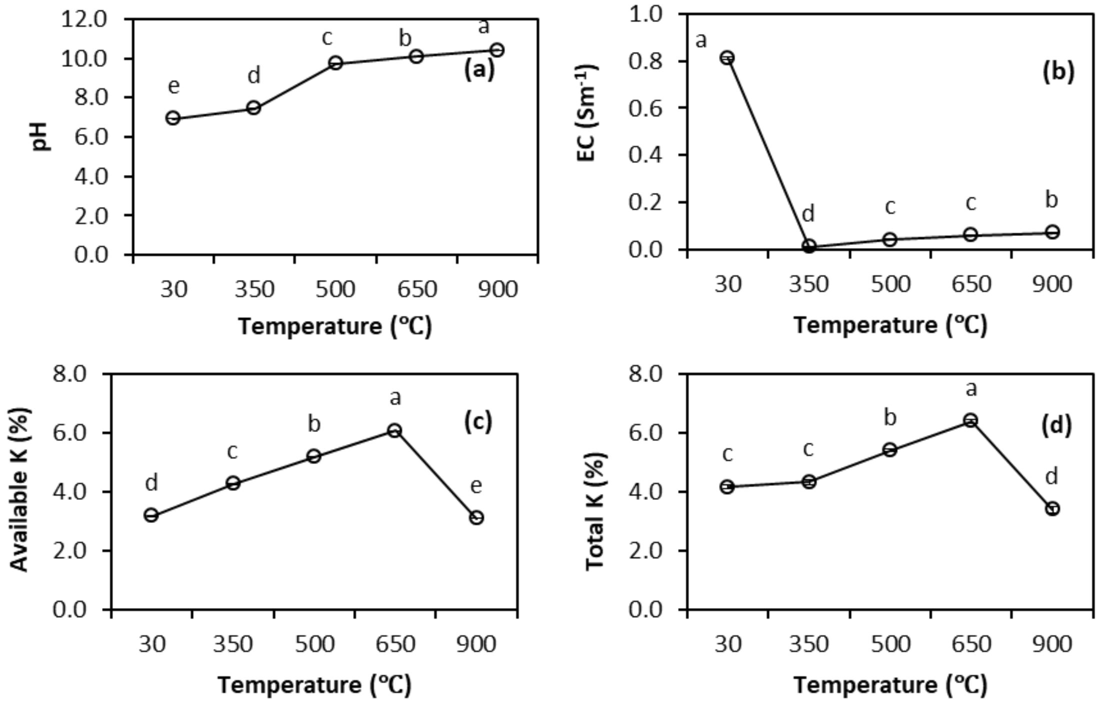

Figure 1. Effect of temperature on pH (a), EC (b), available K (c) and Total K (d) of poultry litter biochar

650 °C. Temperature increased the K availability from 76.2 to 95.7% in pyrolyzed PLB at 650 °C compared that of raw PL (Figure 1c). Berecha et al. (2015) also observed similar results in their experiment using coffee husk and corn cob biochar where available K content and exchangeable cation contents increased when pyrolysis temperatures increased from 350 to 500 °C. Increasing pyrolysis temperature from 300 to 600 °C, increased the total K content of poultry litter biochar from 69.2 to 90.5 g/kg (Song and Guo, 2012). The highest total and available K contents were observed at 650 °C (6.38 and 6.07%, respectively) and were significantly ($p<0.001$) different from others (Figures 1c and 1d). Elevated temperature exceeding 650 °C resulted in a notable reduction in both total and available K contents in PLB (Figures 1c and 1d). The total and available K contents in PLB exhibited a substantial decline from 650 to 900 °C, decreasing by 48 and 47% respectively. At higher pyrolysis temperatures, not only K but also other volatile elements and compounds are released from the biomass. These volatiles can carry away a portion of potassium content, contributing to the observed decrease in total and available K content in the biochar.

Effect of temperature on total K and available K contents in K-bearing minerals

Raw feldspar had significantly higher total K content (6.2%) than mica (4.5%) (Table 1). With the increase in heating temperature, total K content of feldspar significantly ($p=0.003$) increased up to 6.8% until 500 °C and then significantly decreased to 4.6% at 900 °C. A similar trend could be observed in the case of mica as well where total K content was significantly ($p=0.001$) increased up to 5.5% until 500 °C, and then decreased to 1.9% at 900 °C. Feldspar is a potassium aluminosilicate, the general formula being $KAlSi_3O_8$ (Schroeder, 1978). Micas are phyllosilicates containing Si tetrahedra and Al octahedra (Schroeder, 1978). At elevated temperatures, water molecules that are often present as part of the mineral structures can be driven off through dehydration. This lead to increase the total K content in the heated K-bearing mineral samples at 500 °C. The decrease in total K contents above 500 °C could be due to volatilization of K compounds that are not hardly bound to mineral

Table 1. Effect of direct heating temperature on K contents in Feldspar and Mica

Temperature (ºC)	Total K (%)†		Available K (%)†	
	Feldspar	Mica	Feldspar	Mica
30	6.20_{c}	4.50_{b}	0.052_{a}	0.027_{b}
350	6.50_{b}	5.13_{ab}	0.047_{ab}	0.031_{a}
500	6.83_{a}	5.52_{a}	0.042_{b}	0.026_{b}
650	4.63_{d}	3.70_{c}	0.047_{ab}	0.031_{a}
900	4.61_{d}	1.92_{d}	0.031_{c}	0.025_{b}

† Values followed by the same subscript within a column are not significantly different at $p>0.05$

structures (Teixeira et al., 2015; Tumbure et al., 2020). Liou and Wu (2009) observed an establishment of surface vesicles during the heating as a result of the release of volatile compounds inside the matrix of the biochar.

Effect of temperature on total and available K contents in co-pyrolyzed biochar

Addition of K-bearing minerals in the process of biochar production changed total and available K contents of PLBs (Figure 2). There was no significant difference in total K content between PLB_{650} (6.38%) and PLB-$5F_{650}$ (6.27%) or PLB-$5M_{650}$ (6.49%) (Figures 2a and 2b). No notable disparity was observed in the total K content among PLB_{500} (5.40%) and PLB-$5F_{500}$ (6.52%) or PLB-$5M_{500}$ (6.75%). Also, there was no significant difference in total K content between PLB_{350} (4.41%) and PLB-$5F_{350}$ (4.30%) or PLB-$5M_{350}$ (4.58%) (Figure 2a and 2b). These results showed that co-pyrolysis of poultry litter with feldspar or mica did not improve the total K content in PLB at any temperature evaluated in this study. Similarly, co-pyrolysis of PL with K-bearing minerals did not improve the available K content (Figures 2c and 2d). It resulted either in decreased or no change in the availability of K content in PLB at any temperature tested in this study. Compared to available K content in PLB_{650} (6.06%), co-pyrolyzing PL with 5% mica at 650 ℃ (*i.e.* PLB-$5M_{650}$) has decreased the available K content (5.11%) by 19%.

Changes in structural properties of biochar

Scanning electron microscope (SEM) images were used to illustrate the morphological and structural changes of the raw PL, biochar and mineral samples following pyrolysis at 500 ℃ (Figure 3). PL has an intricate porous structure made out of clear inter linked tube-like structures. One of the most noticeable changes when converting raw PL into PLB is the significant increase in porosity. During the pyrolysis, volatile organic compounds and moisture are driven off, leaving behind a carbon-rich matrix with numerous pores. These pores can range from mesopores and macropores (Phuong et al., 2016).

With the increasing pyrolysis temperature, more cracked and enlarged pores can be seen in the pore structure of PLB with or without K-bearing minerals (images not presented). SEM images further indicated that addition of finely ground K-bearing minerals has blocked tube-like pore structures in PLB probably by occupying them with K-bearing minerals. Heating feldspar and mica at 500 ℃ did not show any significant changes in their structures compared to that of unheated minerals. This was also proved by the FTIR spectroscopy results (Figure 4) where temperature did not significantly change the functional groups present in the K-bearing minerals heated at 500 ℃ in comparison with those from the K-bearing minerals without heat treatment.

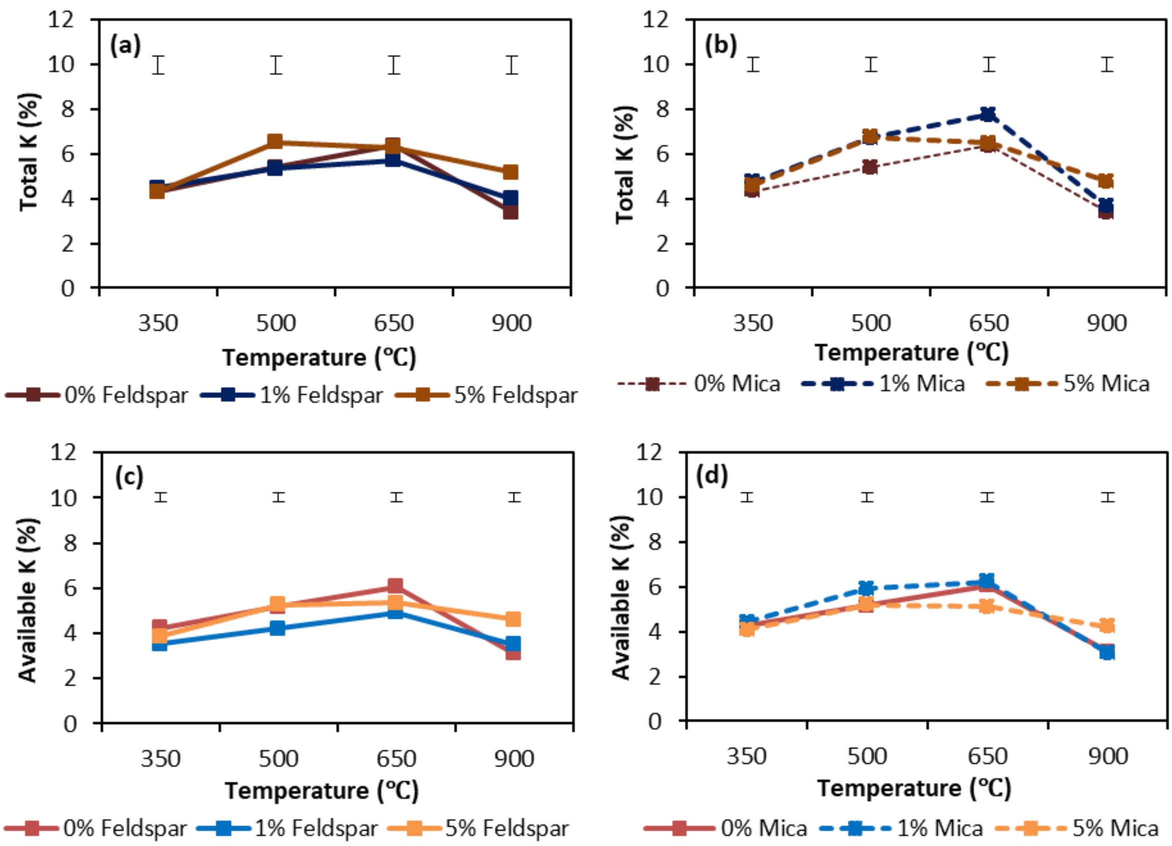

Figure 2. Effect of temperature on total (a and b) and available K (c and d) contents in co-pyrolyzed biochar

FTIR analysis was conducted on PL and samples of PLB that recorded the highest total and available K contents in both minerals (Figure 4). It revealed that K-bearing minerals and co-pyrolyzed PLB at 500 °C had few characteristic peaks within the observed 4000 – 600 cm^{-1} region (Figure 4). There were several new peaks characterized at 1217, 1369 and 1739 cm^{-1} in the PLB500, compared that of raw PL. Peak at 1217 cm-1 is related to esters presents and the spectra of C-O bonds normally appear between 1000-1300 cm^{-1} (Ulusoy, 2020). The visible bands at 1030 cm^{-1} in PL assigned to C–O stretching of polysaccharides (Depeursinge et al., 2010). The observed peak at 1369 cm^{-1} is assigned mainly to CH_2 units in biopolymers that are stable at high temperatures (Chen et al., 2005). The C=O stretching for carboxyl, aldehyde, ketone and ester were identified in the band width 1740–1700 cm^{-1} (Cantrell et al., 2012).

It was observed that similar main peaks at 1217, 1369 and 1739 cm^{-1} in co-pyrolyzed BC and PLB500. But the intensity of stretches at 1217, 1369 and 1739 cm^{-1} have been reduced after co-pyrolysis with K-bearing minerals. Dissanayake et al. (2018) also observed similar results of disappearing or lowering the intensities of several peaks in the biochar made by co-pyrolyzing rice husks with Eppawala Rock Phosphate. Co-pyrolysis of PL with K-bearing minerals at 500 °C has created a new peak at 2330 cm^{-1} which corresponds with triple bonds such as nitriles and carbene groups (Hanandeh et al., 2016). It has been observed that mixing minerals with organic matter just before pyrolysis could increase the discharge of oxygenated compounds including humics and phenolics from biochar (Kleber et al., 2015).

Comparing the FTIR data, it was observed that heat treatment does not significantly affect the morphological changes in the K-bearing minerals but addition of K-bearing minerals prior to co-pyrolysis resulted diminishing of intensities of some prominent stretching vibrations in PLB. There were

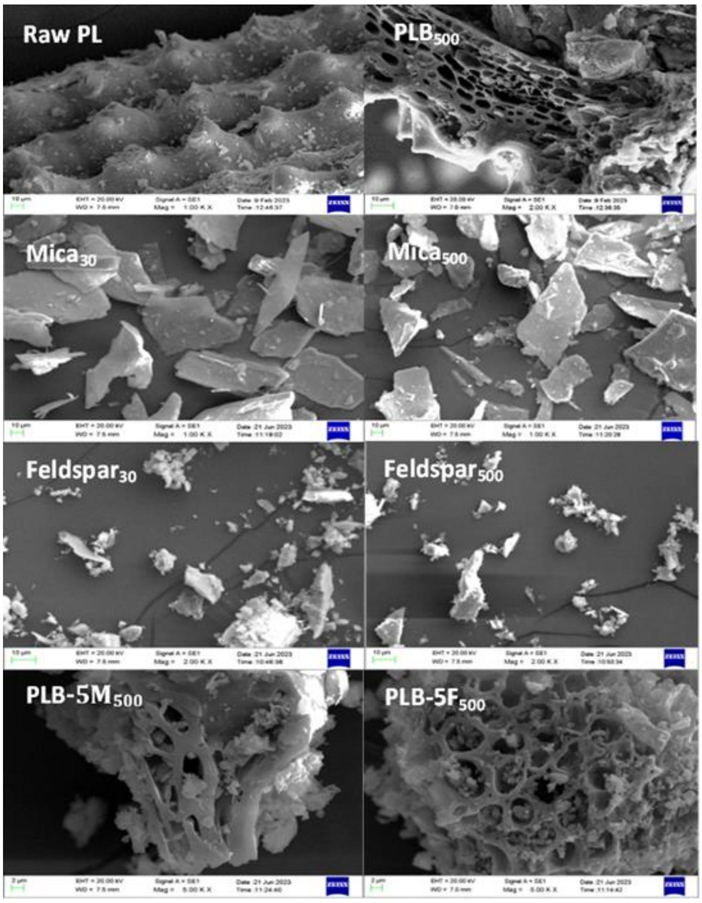

Figure 3. Scanning electron micrographs of raw PL, PLB, K-bearing minerals and co-pyrolyzed biochar produced at 500 ˚C.

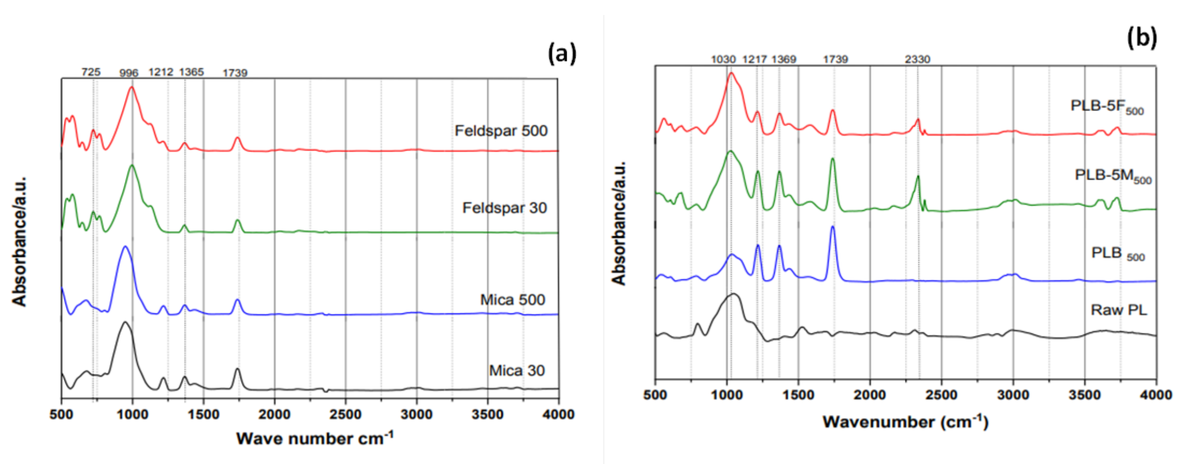

Figure 4. FRIR spectra: (a) Comparison of temperature effect on the K-bearing minerals at 500 ˚C (b) comparison of PL pyrolyzed with and with and without K-bearing minerals at 500 ˚C

peaks observed at 996, 1365 and 1739 cm^{-1} in both K-bearing minerals with and without heat. It was observed that the temperature does not significantly change the functional groups present in the K-bearing minerals heated at 500 ℃ in comparison with those from the K-bearing minerals without heat treatment, suggesting that no new bonds had been created due to heating. Therefore, the observed increased K availability upon heating could be due to the release of K in silicate structures after their break-down.

CONCLUSION

The present study confirms that temperature has a strong impact on total and available K contents in K-bearing minerals, PLB and co-pyrolyzed PLBs. Increasing temperature upto 500 or 650 °C will increase the total K contents in K-bearing minerals, and PLB co-pyrolyzed PLBs and available K contents in PLB and co-pyrolyzed PLBs. Heating raw materials at 900 °C significantly decreases total and available K contents to levels that are lower than their unheated counterparts. Even though heating of locally available Feldspar and Mica minerals up to 500 °C can increase total K contents, heating cannot be considered as a potential technology to increase their effectiveness as a sources of K fertilizer because heating did not significantly increase the available K contents. Co-pyrolyzing PL with K-bearing minerals upto 5% also will not further increase total or available K contents in PLB. Hence it is concluded that co-pyrolysis of PL with locally available K-bearing minerals is not a potential technology to increase the utilization of locally available feldspar and mica minerals as K sources in agriculture.

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