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Morphological and Micro-Grain Structural Analyses of Pretreated Substrates (Sawdust and Poultry Dung) for Potential Use in Enhanced Biogas Production

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ABSTRACT
This study analyzed the morphological and micro-grain structures of pretreated substrates (sawdust and poultry dung) for potential use inenhanced biogas production using Fourier Transform Infrared Spectroscopy (FTIR) and Scanning Electron Microscopy Analysis (SEM). This was done to identify internal microscopic changes that happened because of delignification/pretreatments carried out on the substrates.FTIR and SEM deviceswere used to analyze raw sawdust, chemically (alkaline) pretreated and biologicallypretreated substrates to measure delignification levels. Absorbed radiation was converted into vibrational and rotational energy by molecules and resultant signal was observed at detector end. Samples were also mounted in a chamber and microscope was positioned with scan coils above objective lens.Signals produced were collected by detectors and imageswere displayed onthe monitor.FTIR results ranges for raw sawdust, alkaline pretreated and biological pretreated were 3407.10 - 609.46cm^{-1}; 3452.37 - 572.10cm^{-1}; and 3455.31 - 559.42cm^{-1} respectively. Imagery results from SEM analyses for raw sawdust, alkaline pretreated and biological pretreated sawdust samples were; 2000µm at 16.99mm, 25 display magnitude; 100µm at 15.1mm, 1000 display magnitude; and 100µm at 14.5mm, 750 display magnitude, confirming significant changes in the morphological and micro-grain structures of the substrates. Both alkaline (85.2%) and biological (92.6%) pretreatments on raw sawdust samples decimated the lignin contents, increased the digestibility of the substrates and ultimately, increased their suitability for enhanced biogas production. However, biological pretreatment for raw sawdust yielded better delignification percentage.
Keywords: *Substrates, Pretreatment, Fourier Transform Infrared Analysis, Scanning Electron Microscopy Analysis, Biogas*

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I. INTRODUCTION

Wood is among the most critical renewable resources in the world and this is because they are grown naturally, with little to no human intervention for their survival(Ajayeoba*et al.,* 2021) [1]. More so, they preserve the natural ecosystem because they produce enough clean air to replenish the planet, while also remedying the polluting effects of industrialization(Ajayeoba*et al.,* 2021) [2]. Furthermore, wood is the hard substance made of fibre and composed mostly of branches and stem of a shrub or tree, which is often covered by the tree bark. Similarly, the innermost part of the wood is described as heartwood, while the outer layer of the wood is described as sapwood(Dahunsi*et al.,* 2017) [3]. As such, for industrial and commercial purposes, woods are classified into softwoods and hardwoods. However, the principal chemical components of wood are lignin, hemicellulose and cellulose (Dahunsi, 2019) [4]. Cellulose which is made especially from D-Glucose units is also the critical constituent (40-50%) of both softwood and hardwood. Furthermore, the chemical components of cellulose and lignin are different in hardwood and softwood (Oladejo *et al.,* 2020) [5]. However, the lignin concentration of softwood is better than that of hardwood, which always makes them the choice candidates for pretreatment processes required for substrates in biogas production.

Therefore, raw materials that have very high lignocellulosic concentrations are desirable candidates in the production of biogasas reliable alternatives to fossil fuels (Zumalla*et al.,* 2018) [6].In 2015, livestock manure added nearly 10 percent of all the emissions of methane in the United States of America, still just 3 percent of livestock manure are recycled through anaerobic digesters(United States Environmental Protection Agency, 2009) [7]. When livestock manure is used for the production of biogas, anaerobic digestion can help in reducing nearly 99 percent of manure pathogens, odors and greenhouse gas emissions.It was estimated by the United States Environmental Protection Agency (USEPA)(2009) that there is a possibility for having 8,241 biogas systems powered by livestock manure, which could altogether produce more than 13 million megawatt-hours of energy every year(Ostrem *et al.,* 2014) [8]. The component parts of manure from all the animals are varied, therefore, the manures produced by these animals also vary in their suitability assubstrates for biogas production (Moller *et al.,* 2014), (Budiyono and Sumardiono, 2014)[9], [10].

According to Budiyono*et al.,* (2015), Kunatsa*et al.,* (2013) [11], [12], the manure obtained from cattle generally produces less gas compared with those from poultry and pigs, this is because majority of the organic matter available in the feed are already digested. But if the manure is digested alongside other types of materials like forage crops, food waste and poultry dung, the gas production will improve (Lehtomäki, et al., 2017), (Budiyono*et al.,* 2010) [13], [14]. Consequently, the biological or chemical pretreatment and digestion processes become more enhanced and even sustainable because these organic materials provide the ideal conditions required for the processes. This ultimately culminates in the production of desirable biogas samples desperately needed by developing countries in this century(Börjesson and Mattiasson, 2017)[15]. This can be accomplished through physical methods; (pyrolysis, microwave, mechanical extrusion and milling); chemical methods (acid pretreatment, ozonolysis, alkali pretreatment and ionic liquids); biological methods (brown rot fungi, soft rot fungi and white rot fungi); and physico-chemical methods (steam explosion, ammonia fiber expansion, liquid hot water, carbon dioxide explosion and wet oxidation)(Zumalla*et al.,* 2018), (United States Environmental Protection Agency, 2009)[6], [7].

To this end, this study carried out the morphological and micro-grain structural analyses of pretreated substrates in order to identify their materials orientation, crystalline structure, external morphology and chemical properties, while also justifying the effectiveness of the pretreatment processes carried out on them. This was done in order to bridge the gap of empirical data that presently exists in this research area, especially regarding the selected substrates for pretreatment and their potential use for biogas production.

II. MATERIALS AND METHODS

This section presents the FTIR and SEM techniques adopted for the analyses of the internal structures of the sawdust and poultry dung used for enhanced biogas production in the study.

Step I. FTIR Analysis of the Substrates

The FTIR spectrometer used was the Spectrum 3 Tri-Range MIR/NIR/FIR, manufactured by PerkinElmer, USA, having a wavelength range of 11,000 – 30 cm^{-1}. This was used to send infrared radiation of nearly 10,000 to 100 cm^{-1} through measured representative substrates (sawdust and poultry dung).Furthermore, according to the findings and recommendations of Aftab *et al.,* (2019) [16], the infrared radiation was positioned vertically upright such that some of the targeted radiation was absorbed by the representative samples, while some passed through. The absorbed radiation was converted into vibrational and rotational energy by molecules of the samples. Subsequently, the resultant signal observed at the detector end appeared like a spectrum from 4000 cm^{-1} to 400 cm^{-1}, representing a molecular fingerprint of thesamples. Therefore, every molecule and chemical structure of the samples produced its own unique spectral fingerprint and this was the primary indicator of change that happened to the original composition of the samples.

Step II. SEM Analysis of the Substrates Samples

The SEM used was the Axia ChemiSEM having an inner width of 280 mm, sample weight of 10 kg and was manufactured by Thermo Fisher, USA. This was used according to the findings and recommendations of da Costa et al., 2009) [17]for the examination of the morphology of the samples. The substrates were mounted in the chamber under a combination of pumps. The electron beam of the microscope was positioned on the samples such that they were under the control of the scan coils. Furthermore, the scan coils scanned the electron beam over the surface of the substrates and as the electrons interacted with the substrates, theyproduced characteristics X-rays, backscattered electrons and secondary electrons. Subsequently, the electron beam penetrated the samples deeply as a consequence of the sample density and the accelerating voltage of the electron beam. Eventually, this led to the production of signals which were displayed on the monitor of the computer.

III. RESULTS AND DISCUSSION

This contains and presents the data and results obtained from the laboratory analyses of the internal structures of the sawdust and poultry dung used in this study.

I. FTIR Results

The analyzed values of the FTIR for the raw sawdust, the alkaline pretreated with sawdust, the biological pretreated with poultry dung, alkaline pretreated with poultry dung and the biologicalpretreated sawdust at 15days are presented in Figure 1 to Figure 5 respectively. The values obtained for the raw sawdust in Figure 1 showed that the absorbance band at the frequency end was around 2940.45cm^{-1} and 590.83cm^{-1}, indicating a very high frequency for the raw sample, making it unsuitable for biogas production in the raw state. Hence, the need for the pretreatment processes carried out in this study. Furthermore, these high values obtained are also noticeable in other FTIR results. For alkaline pretreated with sawdust, the value of the absorbance band at the frequency end still remained relatively high at 1632.23cm^{-1} and 580.20cm^{-1}. Similarly, for alkaline pretreated with poultrydung, the values also were 1640.33cm^{-1} and 572.63cm^{-1} respectively.

For biological pretreated with poultry dung, the values obtained from the analysis were 1642cm^{-1}and 572.10 cm^{-1}at the absorbance band and frequency end respectively.However, when biological pretreatment was carried out on sawdust substrates at the 15th day of analyses as shown in Figure 5, the value had fallen significantly down to 1639.14cm^{-1} and 559.42cm^{-1} at the absorbance band and frequency end respectively. These results further indicated that the pretreatment processes carried out on the substrates prior to digestion significantly impacted their digestibility as can be seen in Figure 4 and this also aided the process of biogas production. Therefore, these results confirm the efficacy of both the alkaline and biological pretreatment processes in enhancing the biogas production potentials of the substrates.

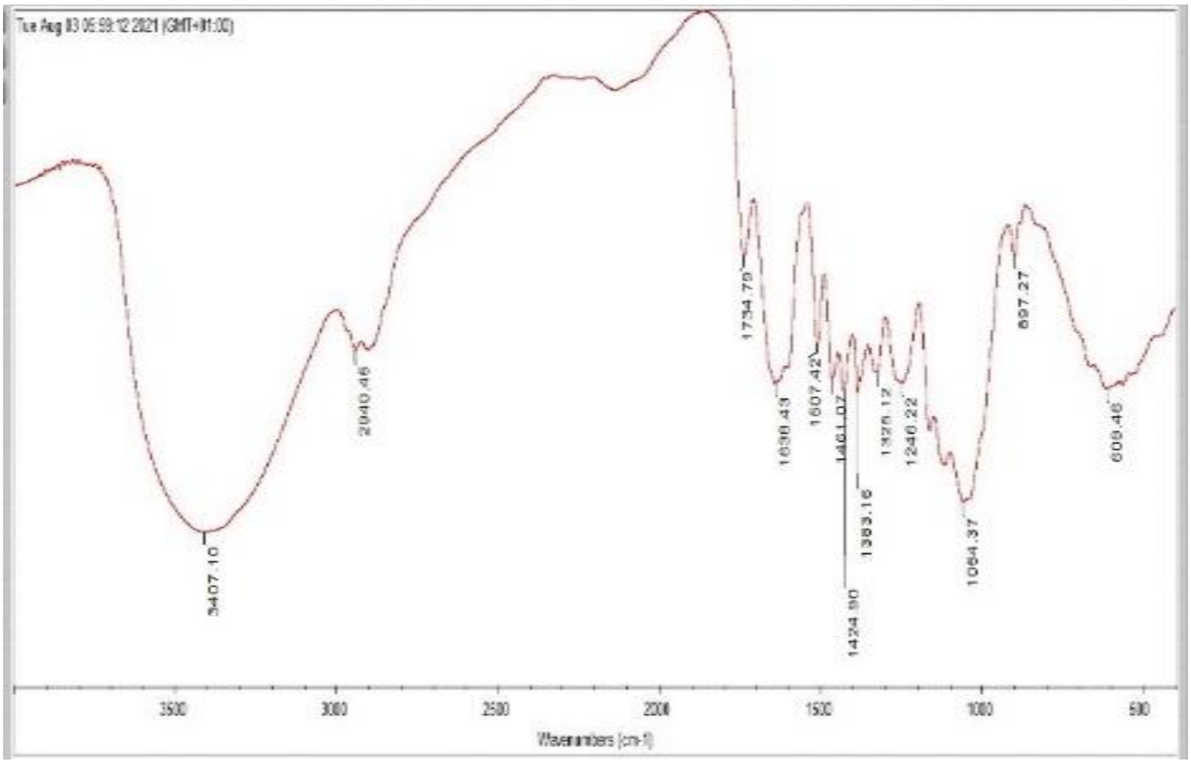

Figure 1: FTIR analyses of raw sawdust samples

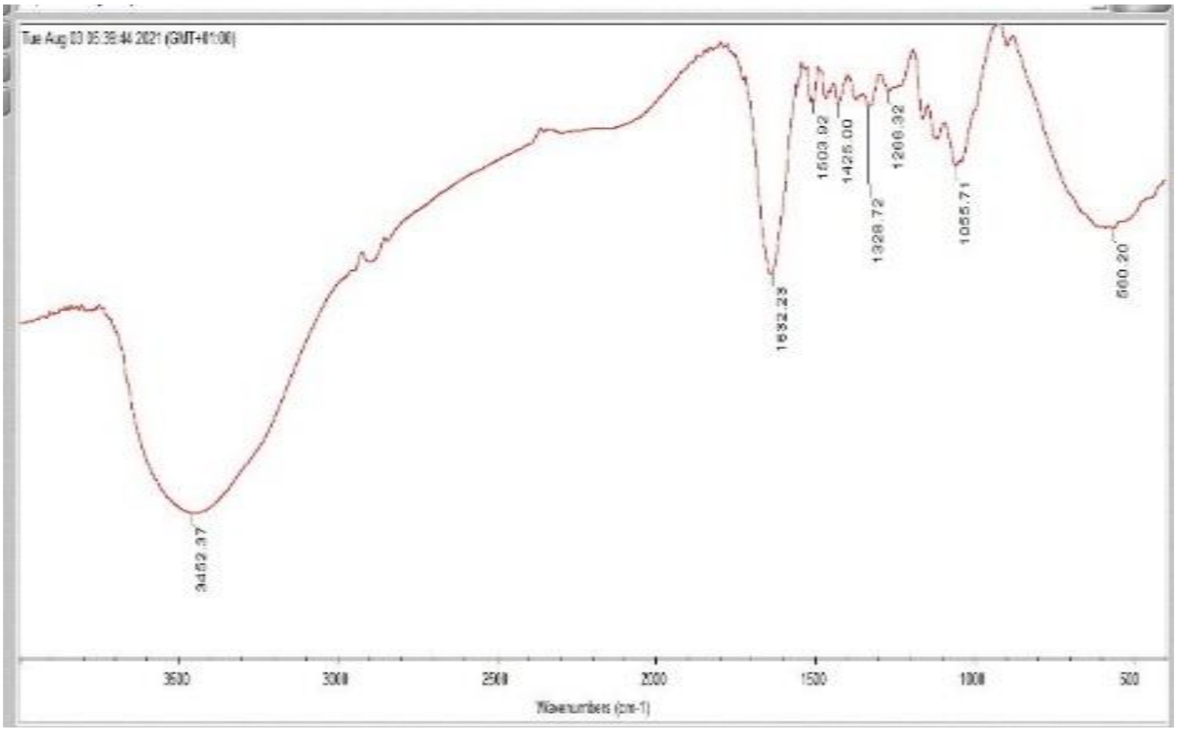

Figure 2: FTIR analyses of alkaline pretreated with sawdust substrates

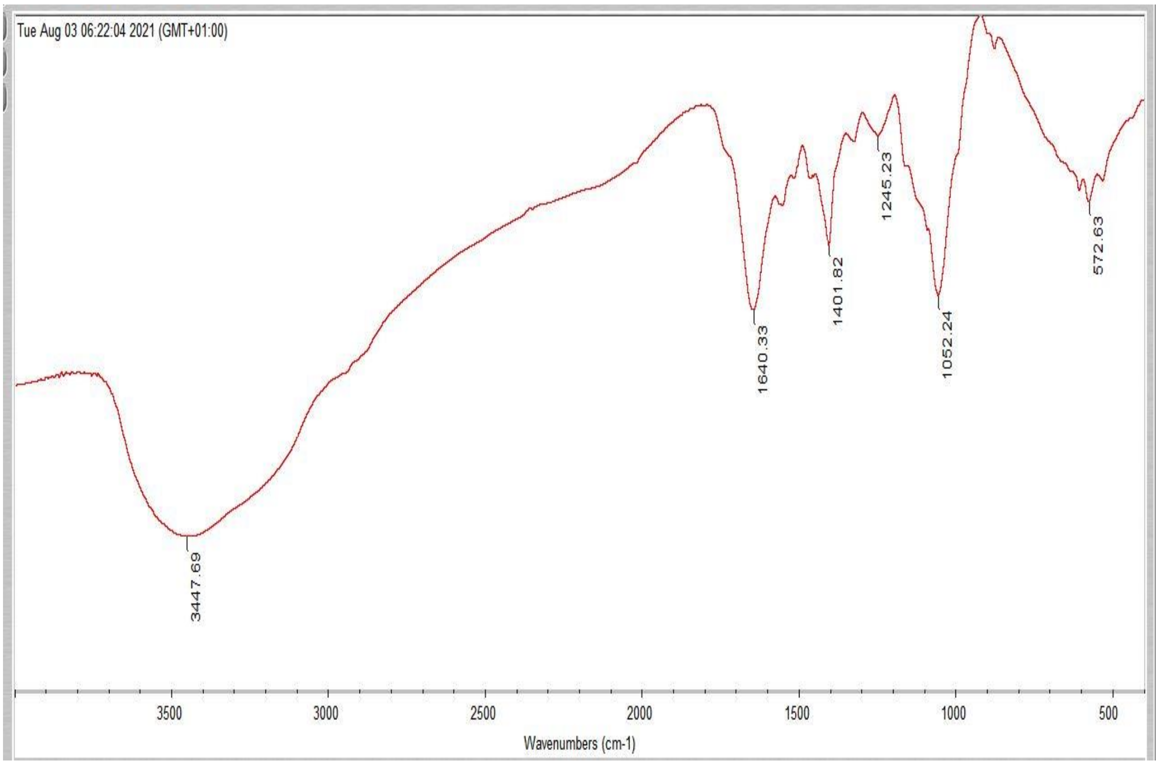

Figure 3: FTIR analyses of alkaline pretreated with poultry dung substrates

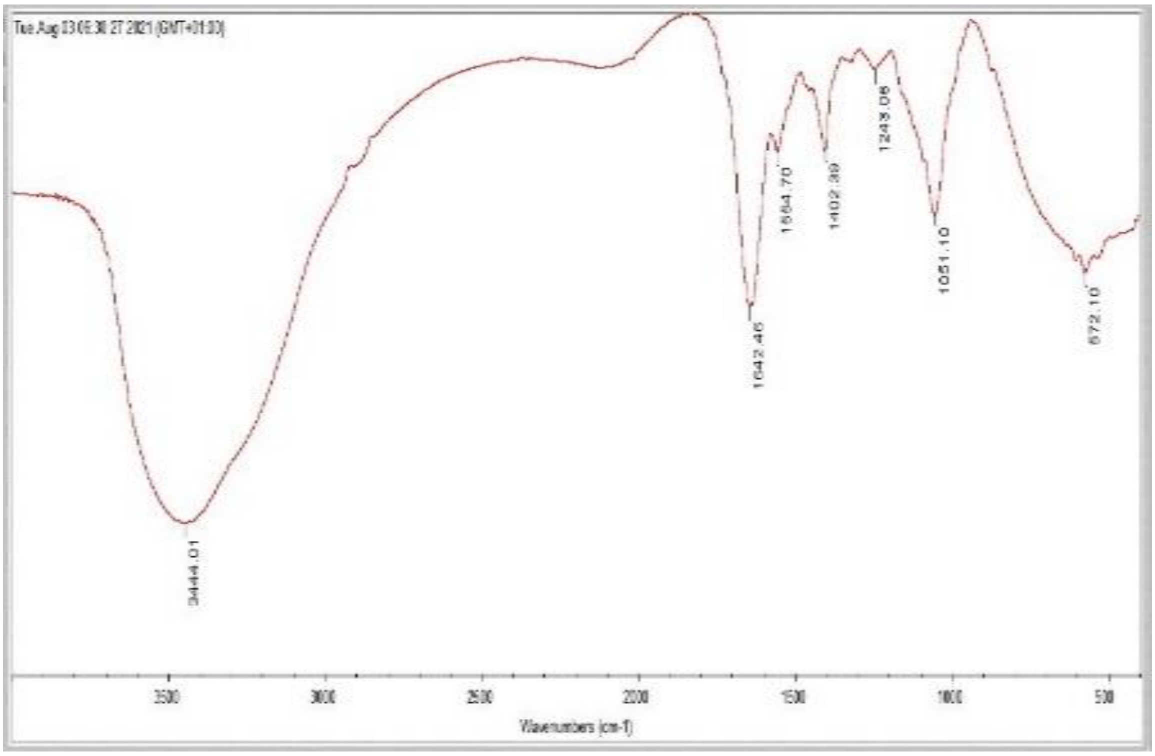

Figure 4: FTIR analyses of biological pretreated with poultry dung substrates

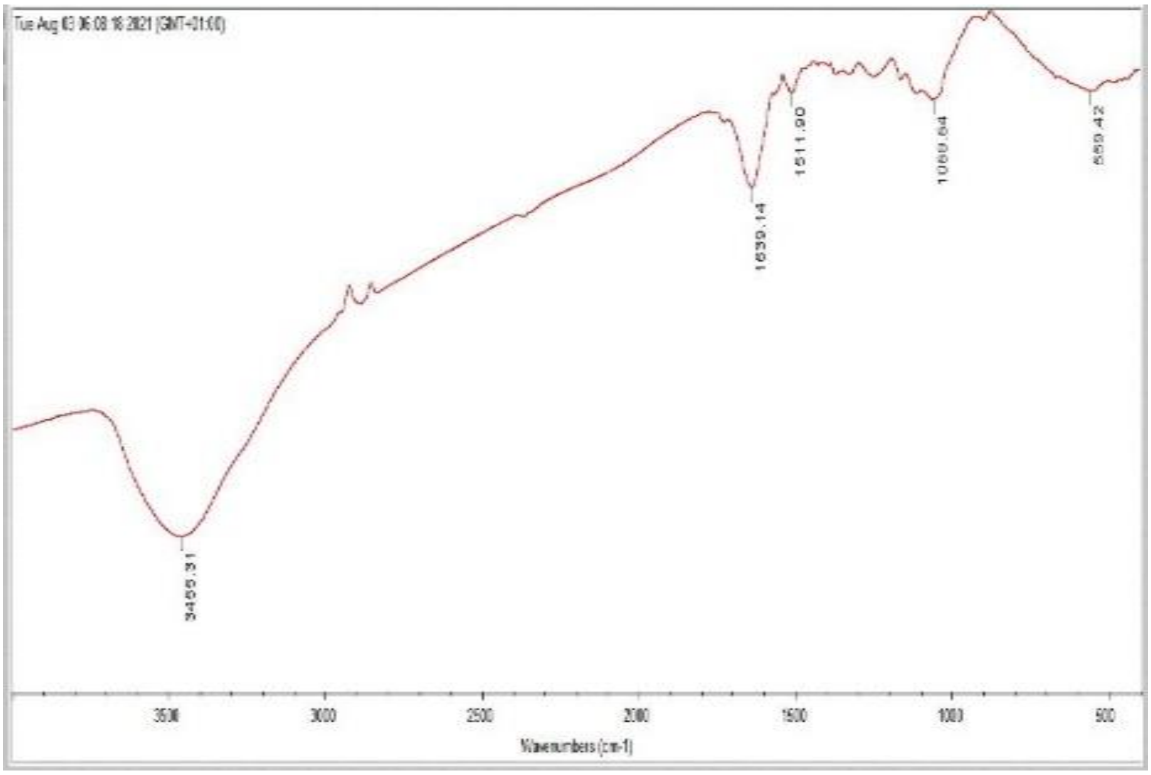

Figure 5: FTIR analyses of biological pretreated with sawdust substrates at 15days

II. SEM Results

The analyzed values of SEM for raw sawdust, alkaline pretreated with sawdust, biological pretreated with poultry dung, alkaline pretreated with poultry dung andbiological pretreated sawdust substrates at 15days are presented in Figure 6 to Figure 10 respectively. The values obtained for raw sawdust as shown in Figure 6 showed that the grain and microstructure of the sample was at 2000µm at 16.99mm and the display magnitude was 25, indicating that the density of raw sawdust was high and indigestible, thereby rendering it unsuitable for biogas production without pretreatment. It also indicated that this value is detrimental for anaerobic digestion and optimal biogas production. This therefore reinforces the need for the pretreatment processes carried out on the substrate samples. These sample density levels, accelerating voltages, display magnitudes and sample microns are also seen in other SEM results as shown in Figure 7 to Figure 10. This is because in Figure 7(alkaline pretreated with sawdust), the values fell significantly to 100µm at 15.1mm, while display magnitude had increased to 1000.

Similarly, in Figure 8 (alkaline pretreated with poultry dung), the grain and microstructure value increased slightly to 200µm at 15.6mm, while display magnitude also fell to 250. Furthermore, in Figure 9(biological pretreated with poultry dung), the grain and microstructure value remained at 200µm at 14.9mm, while display magnitude increased to 500. However, when biological pretreatment was carried out on the sawdust substrates at the 15th day of analyses as shown in Figure 10, the value had fallen down significantly to 100µm at 14.5mm, while display magnitude had increased to 750. These results showedthat the pretreatment processes carried out on the substrates improved sample density values and display magnitude as can be seen in Figure 10. Therefore, these results showed that the pretreatment processeswere efficient in transforming the substrates into useful materials for enhanced biogas production.

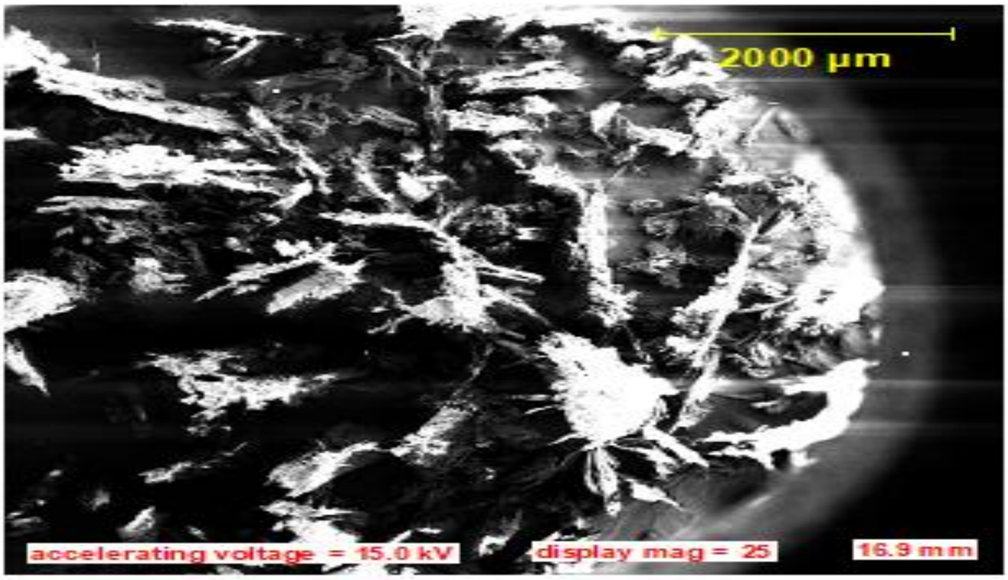

Figure 6: SEM analyzed values of the raw sawdust samples

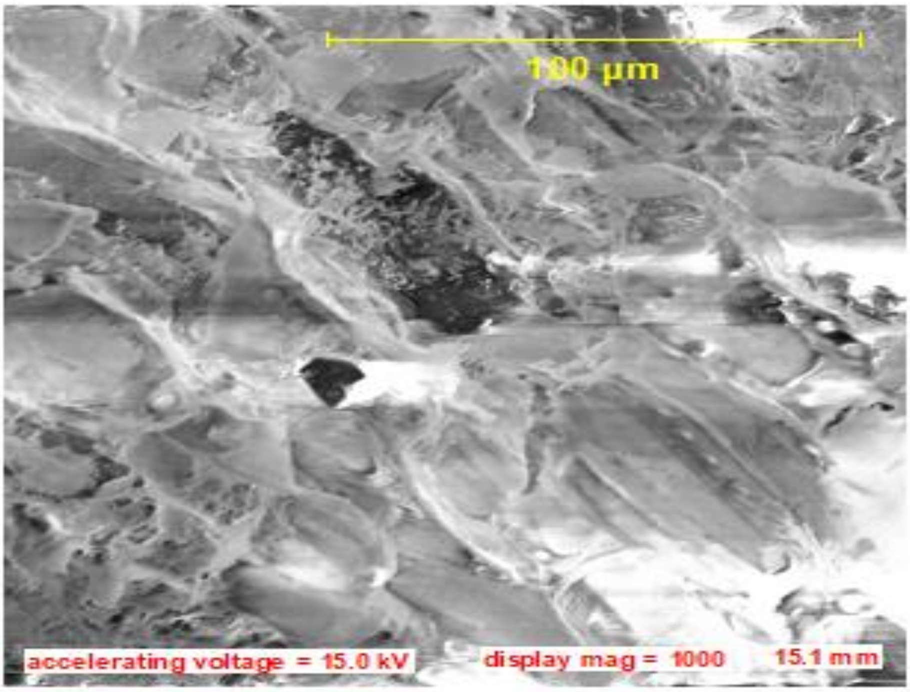

Figure 7: SEM analyses of alkaline pretreated with sawdust substrates

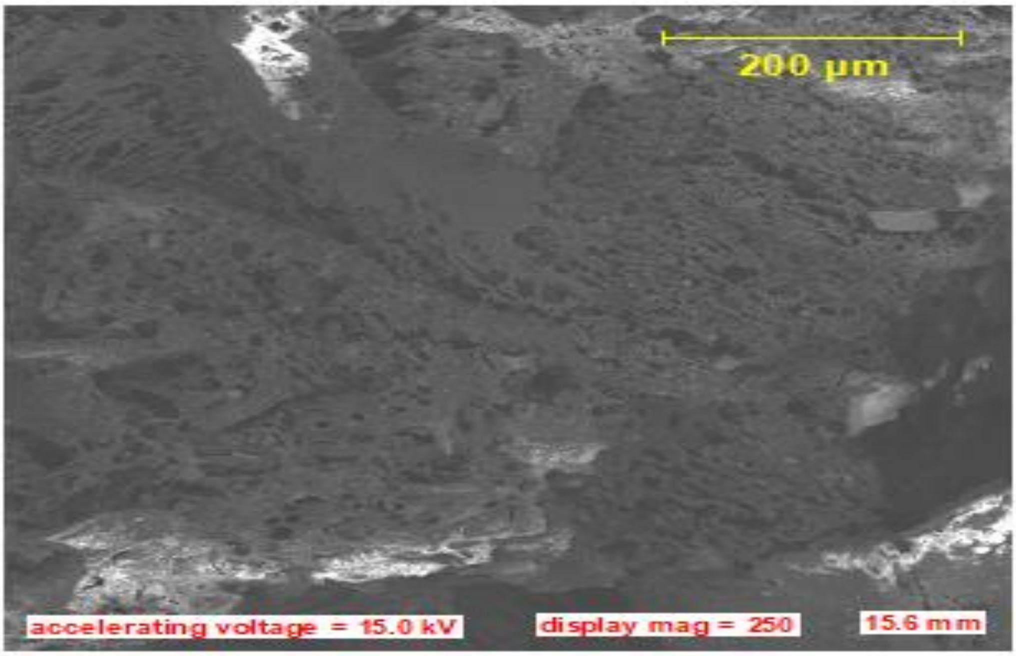

Figure 8: SEM analyses of alkaline pretreated with poultry dung substrates

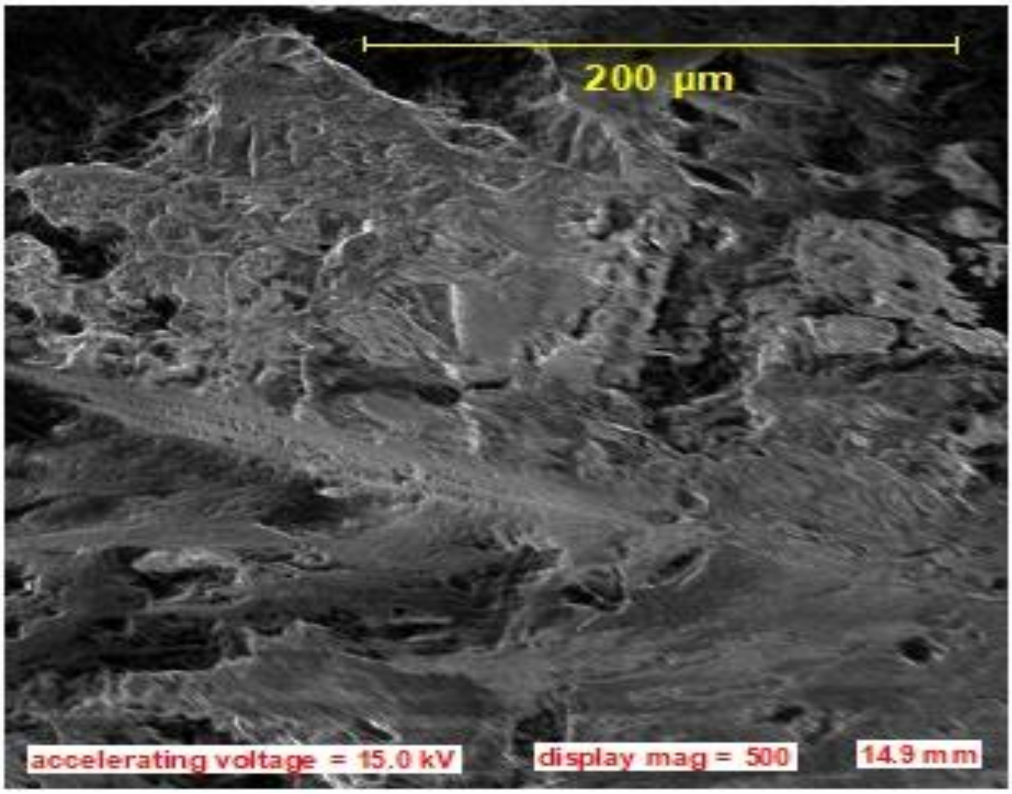

Figure 9: SEM analyses of biological pretreated with poultry dung substrates

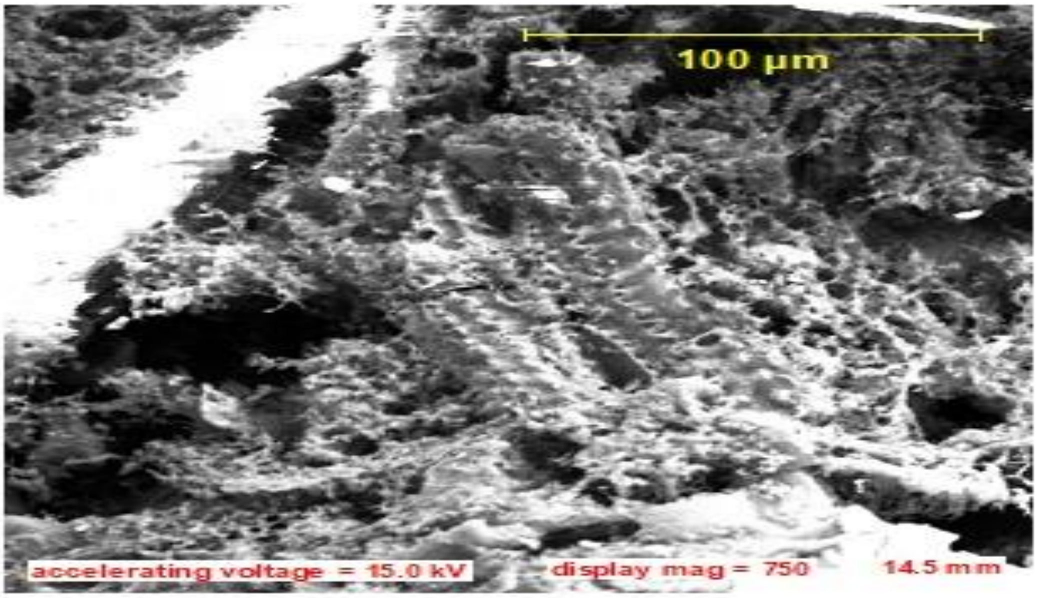

Figure 10: SEM analyses of biological pretreated with sawdust substrates at 15days

IV. CONCLUSION

From this study, it can be concluded that the FTIR and SEM analyses of the substrates showed that on the 15th day of the process, the FTIR values had fallen to 559.42cm-1, while that of the SEM also had fallen to 100µm at 14.5mm and the display magnitude had also increased to 750. The study also concluded that the pretreatment processes conducted prior to digestion had increased the digestibility of the substrates and had thus, increased their suitability for potential use in enhanced biogas production. Furthermore, the biological pretreatment processes conducted prior to the FTIR and SEM analyses produced the better results when compared with the alkaline pretreatment process and therefore, produced better digestibility of the samples analyzed. However, it is important to note that both pretreatment processes performed satisfactorily.

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