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Characterisation of Hydrochar Produced from Poultry Litter and Eggshell Waste by Hydrothermal Carbonisation for Soil Amendment

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ABSTRACT

Converting poultry litter and eggshell waste into a carbon-rich material represents an effective approach. A hydrothermal carbonisation (HTC) technique was employed to produce hydrochar and assess its characteristics to improve soil fertility and promote plant growth. For hydrochar production, poultry litter and eggshell were combined with distilled water at a solid-to-liquid ratio of 1:3 (w/v). The hydrothermal carbonisation (HTC) temperature of 240 °C was maintained throughout, with residence times of 60, 120, and 180 mins. Hydrochars were characterised for physicochemical analysis, Scanning Electron Microscopy (SEM), Fourier Transform Infrared Ray (FTIR) Spectroscopy, and X-ray diffraction (XRD). Physicochemical properties such as bulk density, moisture content, ash content, total carbon, organic matter, pH, EC, N, P, K, Ca, and Mg were higher at 180 mins compared to 60- and 120-mins, except for yield, which was lower at 180 mins. The recovery of (N, P, K, Ca, Mg) at 180 min was 43.72%, 44.39%, 9.83%, 272.80%, and 43.48%, respectively, exceeding those obtained at 60- and 120-mins. The surface morphology analysis revealed that hydrochars had crispy and flaky surfaces with micro- and mesoporous structures, indicating that lignin and cellulose components were denatured during carbonisation. FTIR and XRD analysis revealed differences in structural stability during HTC operation, due to the breakdown of cellulose and hemicellulose and formation of aromatic

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structures at 180 mins. Based on these characteristics, the hydrochar produced can be used as a soil amendment for enhancing the soil fertility of acidic soil and supporting plant growth as a sustainable and environmentally friendly option.

Keywords: Eggshell, hydrothermal carbonisation, hydrochar, poultry litter, soil amendments

INTRODUCTION

The increased growth of the global population resulted in high worldwide demand for food, livestock feed, and the availability of lean meat that is low in cholesterol, which has sparked the rapid growth of the poultry industry, which contributed to the large amount of waste generated (Richa et al., 2020), which can be recycled into valuable materials. Animal waste from livestock farming, such as poultry litter, has become a particular concern due to its high production volume and the significant environmental and ecological challenges it poses, including greenhouse gas emissions, water contamination, and soil pollution (He et al., 2023). In addition to its environmental impact, its revalorization is also important from a socioeconomic perspective because of its fertilising potential, due to its essential nutrient content for agricultural applications (Moreno Racero et al., 2025). The sustainable management goal of this waste is to transform it into value-added products, such as soil amendments or fertilisers, which can be incorporated into the ecological cycle, while concurrently restoring soil physical and chemical properties (Fu et al., 2022; Moreno-Racero et al., 2025).

Poultry production generates vast quantities of litter, manure, eggshells, feathers, and other residues that may present severe environmental pollution and economic challenges when inadequately managed. Valorisation of these wastes provides a sustainable alternative to conventional disposal by enabling the recovery of nutrients and mineral components for beneficial applications (Zinina et al., 2022). Poultry litter can be utilised as an organic soil amendment, while waste eggshells, which are predominantly composed of calcium carbonate, can be converted into calcium-rich materials for agricultural, environmental, and industrial applications (Awogbemi et al., 2022). Appropriate utilisation of these poultry-derived wastes can reduce environmental pollution, improve nutrient recycling, enhance soil quality, and support sustainable agricultural practices (Thyagarajan et al., 2013). Therefore, converting poultry litter and eggshell waste into value-added products represents a practical approach to waste management and resource recovery within a circular economy framework.

Traditional thermochemical techniques, such as pyrolysis at high and low temperatures, are the main methods used to produce carbon-rich materials that are used for soil amendments from organic waste. Whereas in thermal treatments under limited-oxygen conditions, the first pyrolysis uses dry organic waste at temperatures between 300 °C and

700 °C (Moreno-Racero et al., 2025). The product is called biochar and is used for soil applications. For the biomass materials that are wet, conventional thermal drying is often needed that sometimes economically unfeasible due to the energy required and the time required (Wang et al., 2018).

To overcome the above-mentioned limitation, hydrothermal carbonisation (HTC) has emerged as another thermal treatment for waste biomass, which has recently received increasing attention as it produces a carbon-rich material called hydrochar. Hydrochar could be used as a soil amendment to enhance soil physical and chemical properties and improve plant growth (Huang et al., 2023). It has also been reported to be a cost-effective, carbon-rich material that is environmentally friendly (Wang et al., 2025). Hydrochar, when applied to soil, provides a circular nutrient cycling medium by returning nutrients from low-cost biomass to the earth (Wang et al., 2020). Hydrochar contains a substantial number of major elements such as C, N, P, and other minerals that can increase soil fertility and promote carbon sequestration, as reported by Belete et al. (2021). Hydrochar significantly improves soil physical-chemical properties and microbial communities that are very important in agriculture by directly or indirectly supplying essential nutrients (Wang et al., 2025). The HTC process produces the so-called hydrochar at temperatures between 150 °C and 350 °C in the presence of water, under its autogenous pressure (Cavali et al., 2023).

The importance of converting wet biomass, poultry litter (PL) and Eggshell (ES), into hydrochar, instead of biochar, is primarily due to the lower production costs associated with it, reduced energy requirements and the elimination of the need for pre-drying the biomass (Paneque et al., 2019). Soils enriched with hydrochar also have a lower biochemical reaction than soils treated with biochar. The result makes it easier for nutrients that were stabilised during the HTC treatment to be remobilised into forms that plants can use. However, it has been observed that during the HTC process, water-soluble components are released into or removed with the process water (Moreno-Racero et al., 2025). This can lead to the loss of beneficial plant nutrients, such as nitrogen (N), phosphorus (P), and other essential minerals, alongside potentially phytotoxic compounds (He et al., 2023). However, plants can utilise a significant amount of nutrients, as hydrochar has the capacity to retain them.

Poultry litter (PL) and Eggshells (ES) are known to be poultry and food industry waste, consisting of macronutrients, organic materials, and trace minerals. Eggshells, often regarded as waste from kitchens and the food industry, are primarily made up of calcium carbonate, different organic materials, and microminerals, in which hydrochar production from them results in a product rich in calcium content that can boost the fertility of the soil and promote plant growth and nutrient availability. Additionally, its porous nature improves aeration, rendering it an important soil amendment. The use of eggshells not only creates an innovative process for reusing shells but also offers significant benefits for sustainable agriculture and environmental cleanup (Wang et al., 2025). Eggshell is one of the liming materials that is applied for the improvement of soil pH, available P, and exchangeable

cations, and it can also be used to decrease the concentration of heavy metals (Almaroai et al., 2014). High calcium, magnesium and phosphorus content from eggshells makes it reusable as a fertiliser or soil amendment (Bashir & Manusamy, 2015). Mixing of PL and ES to get a carbon-rich material known as hydrochar is important in soil amendments. Treatments of these wastes at higher temperatures and pressures using a water medium, through hydrothermal carbonisation, have resulted in the production of a solid product with high carbon content and improved nutrient retention properties. High porosity, low density, and different surface chemistry are some of the unique characteristics possessed by hydrochar, making effective soil amendment that can enhance soil structure, moisture retention, and improve plant growth (Wang et al., 2025). Hydrochar can be used as a source of energy through combustion, an adsorbent for heavy metals and organic pollutants, and for soil amendments (Mau et al., 2020). It is also reported that hydrochar application enhances nutrient availability and reinforcement of soil-carbon structural stability (Vieira et al., 2026). Therefore, knowledge about the most suitable methods for transforming poultry litter and eggshell waste into thermally treated products appropriate for use as soil amendments to improve crop production is still limited. Considering this scenario and to address these knowledge gaps, this study investigated hydrochar production through hydrothermal carbonisation and evaluated its potential as a soil amendment to enhance soil fertility and promote plant growth.

MATERIALS AND METHODS

Raw Materials

The poultry litter was sourced from D'Khairan poultry farm located at Sungai Besar, Selangor, and eggshells were sourced locally from an open restaurant kitchen, all in Malaysia.

Biomass Preparation

The poultry litter collected were separated from debris, shredded and ground into pieces, while the eggshells (ES) were washed thoroughly with distilled water to remove impurities, crushed by mortar and pestle, oven-dried, and milled using a blender. The biomass was dried at 105 °C for 24 hr and subjected to particles of decreasing diameter openings. Manual agitation using a sieve was used to get enough of the desired particle sizes. The samples were then stored in an airtight plastic container before the hydrochar production.

Production Method

The hydrochar was produced following the method described by Bento et al. (2019) as shown in Figure 1. The ground poultry litter and eggshell powder were mixed at a ratio

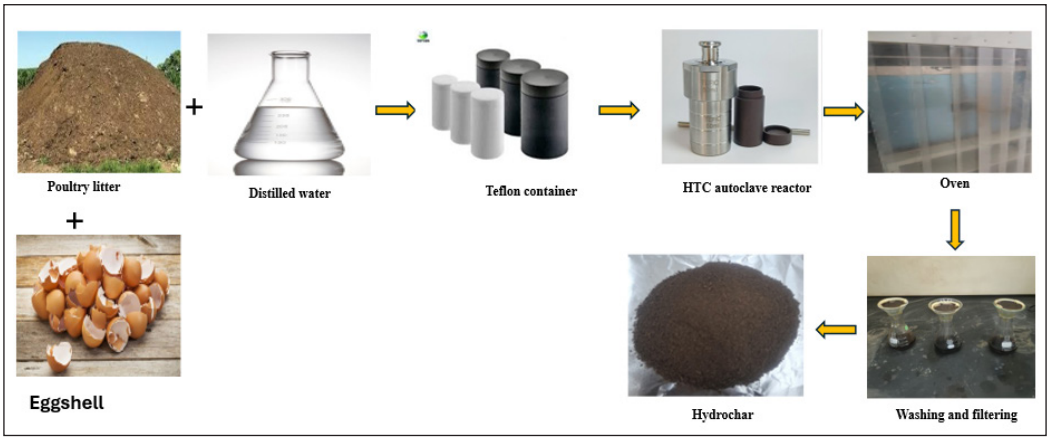

Figure 1. Graphical summary of the multi-step hydrothermal carbonisation process for transforming poultry litter and eggshell into hydrochar

of 50:50 (w/w). After which the resulting mixture was combined with distilled water at a solid-to-liquid ratio of 1:3 (w/v) and transferred into a 100 mL non-stirred Teflon-lined reactor suitable for hydrothermal treatment. The reactor was sealed within a stainless-steel autoclave and subsequently placed in an oven for the hydrothermal carbonisation process. The hydrothermal carbonisation (HTC) temperature of 240 °C was maintained throughout and a reaction time of 60, 120, and 180 mins for the biomass mixtures was employed; it took about an hour of heating time to stabilise the desired heating temperature, and all the experiments were conducted in triplicate to improve the reliability of the data and reduce the effects of random experimental error. After carbonisation, the autoclave reactor was allowed to cool down to room temperature before opening the Teflon container. The carbonised products were separated from the liquid phase using a Whatman filter paper No. 2 to obtain the solid hydrochar. The hydrochar was washed with distilled water during the separation to remove the organic components and subsequently oven-dried at 80 °C for 6 hr. The final dried hydrochar product was stored in an air-tight plastic bag for further analysis

Characterisations Method

Analytical techniques used for the structural composition were Scanning Electron Microscopy (SEM), X-ray Diffraction (XRD), and Fourier Transform Infrared Red (FTIR) spectroscopy.

X-Ray Diffraction (XRD)

The X-ray diffraction (XRD) analysis of hydrochar produced at different residence times was conducted using a Rigaku MiniFlex600-C X-ray Diffractometer, Japan. The prepared

powder samples were placed in a sample holder. Thereafter, the sample holder was arranged in the diffractometer operated at 40 kV voltage, and the sample was scanned from 20-100° with a speed of 1° per minute.

Scanning Electron Microscopy (SEM)

Field Emission Scanning Electron Microscopy (FEI Nova Nanosem 230 FESEM, Netherlands) was used to determine the surface morphology of the raw biomass and the resulting hydrochar. The voltage at which the machine was used was accelerated from 0.2 to 30 kV. The high image resolution was achieved by the high-vacuum conditions with the use of an in-lens secondary electron detector, which gave a spatial resolution of about 1.0 nm at 15 kV. Before the analysis, the samples were placed on the aluminium sheets and then coated with a thin conductive layer.

Fourier Transform Infrared (FTIR) Spectroscopy

Determination of the functional group in each sample was done using an FTIR analyser (Bruker; Invenio R; Germany). The wavenumbers for each sample were measured in the range 4000 to 400 cm^{-1} using 32 scans and a resolution of 4 cm^{-1}. Each sample was ground in a mortar and pestle to produce a fine powder. About 1g of the dried sample was taken for analysis.

Physical Properties Determination

The physical properties of the hydrochar, such as bulk density, moisture content, and ash content, were determined according to the standard procedure. Bulk density and ash content were determined following the method described by Pavkov et al. (2022). The ash content was measured on a dry basis, where about 5g of dried hydrochar sample was weighed and ashed in the muffle furnace at 550° C for 4 to 5 hrs until the sample became completely white. The difference in mass before and after the ashing represents the ash content of the hydrochar. Bulk density of the hydrochar was measured using a cylinder with a defined volume, where the dried and ground hydrochar was filled into a glass cylinder (100 cm^3) by gently tapping it against the table. The excess amount on the top of the cylinder was removed with a stirring rod sliding along the top edge of the cylinder, and the sample mass was then measured. Bulk density was measured by dividing the mass of the sample by volume. The moisture content was determined by Peters et al. (2003), in which the samples were oven-dried at 105° C until a constant weight was reached.

Elemental Composition

The chemical composition of the corresponding hydrochars was determined following a standard procedure. pH and EC of the hydrochar samples were determined using the

method by Lee et al. (2013), where 5g of the sample was weighed into a plastic vial and mixed with 50 mL of distilled water. The plastic vial was covered with a lid and shaken for 1 hr, after which it was allowed to settle for another 1 hr before the reading was taken using the pH and EC meters, respectively. Total carbon, hydrogen, nitrogen, and sulphur contents were determined using the CHNS 628 analyser (Leco®, USA) (Dumas method), and other elements (P, K, Ca, and Mg) were determined using the dry ashing method (Cottenie, 1980). The total (P, K, Ca, and Mg) from the hydrochar were determined using the dry ashing method (Cottenie, 1980). 0.5 g of ground hydrochar samples were weighed and put into a crucible, after which they were transferred into a muffle furnace to be ashed at an initial temperature of 300 °C for 1 hr, and then the temperature was subsequently increased to 500 °C for about 4 to 5 hrs. It was then allowed to cool down in the desiccator; the samples were then placed in a fume cupboard, and a few drops of distilled water were added to the ash samples, followed by the addition of 2 mL concentrated HCl, and were then allowed to evaporate to dryness on a hot plate. Subsequently, 10 mL of 20% HNO_3 (200 mL HNO_3 in 1L distilled water) was added to the samples and was then placed in a water bath for I hr. The samples were transferred into a 100 mL volumetric flask and made up to volume with distilled water before filtering into a 100 mL plastic vial using Whatman No. 2 filter paper. The concentration of P was determined using an auto-analyser machine (AA), while K, Ca, and Mg were determined using an Atomic Absorption Spectroscopy machine (AAS). Cation exchange capacity (CEC) was measured by a comprehensive exchange method based on Gillman and Sumpter (1986)

Determination of Nutrient Recovery

The Nutrient recovery into hydrochar was calculated using the Equation 1 below, as described by Devnath et al. (2024)

$$Nutrient\ recovery\ (\%) \frac{Hydrochar\ yield\ \times\ Hydrochar\ nutrient\ content}{Feedstock\ nutrient\ content} \times\ 100 \quad [1]$$

Statistical Analysis

The data were analysed using SAS software (version 9.4), and the effects of different treatments were separated by Duncan's multiple range test, which was carried out to establish significant differences between the means ($p \leq 0.05$).

RESULTS AND DISCUSSION

Characteristics of Poultry Litter and Eggshell Waste Mixtures

Table 1
Selected physico-chemical properties of poultry litter and eggshell waste mixtures used for hydrochar production

Parameters	Value Obtained
Bulk density (g/cm^3)	3.75
Moisture content (%)	40.14
Ash content (%)	58.15
Total Carbon (%)	24.27
Organic Matter (%)	41.85
pH	8.45
Electrical conductivity (mS/cm)	3.38
Nitrogen (%)	0.90
Phosphorous (%)	0.52
Potassium (%)	1.50
Calcium (%)	14.60
Magnesium (%)	0.48
Cation exchange capacity ($Cmol_+$/kg)	6.95

The combined mixture of poultry litter and eggshell used in the study has a bulk density of 3.75g/cm^3 and a moisture content of 40.14%, which indicates its suitability for hydrothermal carbonisation. Feedstock that has a moisture content ranging from 75 to 90% has been reported to be ideal for hydrothermal carbonisation (Hejna et al., 2022). The proximate results of other physical and chemical properties of the raw feedstock used in this study are presented in Table 1. Organic matter, total carbon content and other elements like K and Mg were lower than the reported range from previous studies (Bernal et al., 2024; Maruthamuthu et al., 2024), but all other physical and chemical properties obtained were within the reported range of values. The 0.90% N, 0.52% P, and CEC of 6.95 cmol(+) kg^{-1} indicate its suitability for use as a nutrient-rich material for hydrothermal carbonisation. The nitrogen content is primarily derived from protein residues and other organic compounds present in poultry litter, while phosphorus is contributed by both organic and mineral components. The addition of eggshell introduces calcium-rich mineral phases, mainly in the form of $CaCO_3$, which may affect the mineral composition and cation exchange properties of the feedstock (Awogbemi et al., 2022). The observed CEC value indicates the presence of exchangeable cations and negatively charged functional groups on the material surface, which play an important role in nutrient retention and transformation during HTC (Wang et al., 2018).

The moisture content of the sample, at 40.14%, exceeds the specified range for raw poultry litter, which is 8.5% to 38.8%. The elevated moisture content is beneficial for HTC, as the process uses water as a reaction medium and does not necessitate pre-drying, in contrast to traditional pyrolysis. The pH value of 8.45 signifies that the poultry litter is mildly alkaline. This alkalinity arises from the decomposition of uric acid and the presence of calcium carbonate from dietary supplements (Risberg et al., 2017). This feature indicates that the resultant hydrochar may function as a liming agent, helping in the mitigation of soil acidity and enhancing nutrient accessibility in acidic soils. The electrical conductivity of 3.38 mS/m, falling within the literature range of 1.47-10.05 mS/m as reported by

(Maruthamuthu et al., 2024), indicates the existence of soluble salts and exchangeable cations, including K^+, Ca^{2+}, and Mg^{2+}, which are advantageous for plant growth when administered at suitable levels.

Effects of Residence Time, Product Yield, and its Properties

The effects of different residence times (60, 120, and 180 minutes) on the product yield and other physical properties of hydrochar produced through HTC are presented in Table 2. There were significant differences ($P \leq 0.05$) between the different times.

Table 2
Mean (± s.e.) effects of different processing times (60, 120, and 180 minutes) on the product yield and other properties

Temperature	Time (Mins)	Yield (%)	Bulk density (gcm^{-3})	Moisture content (%)	Ash content (%)	Organic matter (%)	Carbon (%)
240 °C	60	79.31±0.006[a]	0.38±0.014[c]	67.89±0.338[c]	62.89±0.672[c]	29.20±0.333[c]	17.39±0.338[c]
	120	73.97±0.003[b]	0.43±0.021[b]	69.01±0.662[b]	65.13±0.667[b]	36.36±0.028[b]	21.54±0.328[b]
	180	71.00±0.557[c]	0.49±0.012[a]	69.51±0.328[a]	72.28±0.026[a]	38.57±0.105[a]	22.86±0.095[a]

Note. Means with the same letter in the same column are not significantly different by the Duncan multiple range test at $p \leq 0.05$

Yield and Bulk Density

There was a clear inverse relationship noticed between the processing time and product yield. The hydrochar yield decreased significantly from 79.32% at 60 mins to 71.00% at 180 mins, representing a reduction of 8.32% points, equivalent to a 10.49% decrease relative to the initial 60 mins yield Table 2. This decrease indicates longer thermal conversion processes, in which prolonged residence time enhances the devolatilization of light organic compounds and promotes secondary decomposition reactions, resulting in greater mass loss (Wang et al., 2021). A temperature of 240°C combined with a longer residence time of 180 mins result to greater decomposition, fragmentation, and dissolution of organic waste. The observed decrease in hydrochar yield could be attributed to the progressive decomposition of hemicellulose, lignin, and, to a lesser extent, cellulose in the raw material under hydrothermal conditions (Sabry et al., 2024). The reduced hydrochar yield is most likely due to ongoing dehydration during the hydrothermal carbonisation

process at prolonged residence times, and the removal of oxygen and hydrogen from the hydrochar as the hydrochar becomes more coalified (Azaare et al., 2021; Mau et al., 2020). Bulk density showed a significant positive trend, increasing from 0.38 to 0.48 gcm^{-3} that account for 26.32% increase with longer residence time. The increase in bulk density from 0.38 to 0.48 g cm^{-3} as residence time increased corresponds to a 26.32% rise and may indicate gradual structural rearrangement and consolidation of the carbonaceous matrix during hydrothermal treatment. Extended treatment may enhance the decomposition of labile components, condensation of carbonaceous structures, rearrangement of particles, and alterations in interparticle void volume, thereby influencing the packing characteristics of the hydrochar. Similar increases in density with longer hydrothermal treatment have also been reported for biomass-derived carbonaceous materials (Hussain et al., 2024; Marrocchi et al., 2024). This densification is likely because of the loss of volatiles and moisture, resulting in a more consolidated and energy-dense solid product. High bulk density is an important characteristic for solid biofuels, as it improves handling, storage, and transportation efficiency (Chen et al., 2021).

Proximate Composition: Moisture, Ash, Organic Matter, and Carbon

The effect of different residence times on moisture, ash, organic matter and carbon is presented in Table 2, which shows a slight but significant increase over time, rising from 67.89 to 69.51%, corresponding to an absolute increase of 1.62% points and a relative increase of approximately 2.39%. This trend could be explained by the nature of the process (e.g., hydrothermal treatment), where bound water is retained or even reformed within the polymeric matrix of the biomass as hemicellulose and cellulose degrade (Li et al., 2022). The values of the moisture content were also in line with the report of Mau et al. (2020), who found the moisture content from the poultry litter hydrochar to be 74.05%, 69.48%, and 50.88% for 180, 220, and 240 °C hydrothermal carbonisation temperatures. The Ash content value increased from 62.89 to 72.28%, representing an absolute increase of 9.39% point and a relative increase of nearly 14.93% at 180 mins residence time and 12.99% decrease under 60 mins residence time. This study shows a concentration effect; as organic matter volatilizes, the inorganic mineral fraction (ash) becomes a larger proportion of the remaining solid. High ash content can be a setback for thermochemical energy conversion due to slagging issues, but may be beneficial for applications such as soil amendment, where minerals contribute to nutrient supply (Niu et al., 2019). Organic matter and carbon content both increased significantly with processing time. Organic matter rose from 29.20 to 38.57%, which accounted to 32.09% increase for 60 mins to 180 mins residence time, while carbon content increased from 17.39 to 22.86%, which showed about 31.45% increase from 60 mins to 180 mins hydrothermal carbonisation time. This shows that prolonged treatment residence time promotes carbonisation, enhancing the relative abundance of

stable, carbon-rich structures in the solid product. On the other hand, carbon content in hydrochar products increases significantly. Higher carbon content is mainly as the result of the carbonisation process that occurs during HTC. Furthermore, carbon content tends to rise with higher reaction temperatures and significantly changes with prolonged residence times, confirming that both temperature and time are important factors in the carbonisation process (Sabry et al., 2024). This aligns with established biochar production principles, where extended residence time promotes aromatisation and carbon stabilisation (Lehmann & Joseph, 2024).

Potential hydrogen (pH) and Electrical Conductivity (EC)

The pH and EC of the hydrochar are presented in Table 3. There was a significant increase in pH from 8.63 to 8.77 with prolonged processing time, indicating progressive alkalization. The pH value progressively increased with increasing residence time, rising from 8.63 at 60 mins to 8.72 at 120 mins, corresponding to a 1.045 increase. A further increase from 8.72 at 120 mins to 8.77 at 180 mins was observed, representing an additional 0.5% increase. Generally, the value increased from 8.63 at 60 mins to 8.77 at 180 mins, relating to a total relative increase of nearly 1.62%. This is ideal in thermal conversion processes like pyrolysis or hydrothermal carbonisation, in which the alkaline salts (carbonates, oxides) are formed and concentrated as volatilized organic acids (Wang et al., 2021). Electrical conductivity (EC) also increased significantly from 1.64 to 2.69 $mScm^{-1}$, the EC value increased significantly with increasing residence time, from 1.64 at 60 mins to 2.06 at 120 mins, accounting for a 25.61% increase. It then increased further from 2.06 at 120 mins to 2.69 at 180 mins, resulting in an additional 30.58% increase. Overall, the value rose from 1.64 at 60 mins to 2.69 at 180 mins, representing a total relative increase of approximately 64.02%, indicating a substantial rise in soluble ion concentration that could be attributed to the release of mineral elements (K^+, Ca^{2+}, Mg^{2+}, etc.) and the formation of insoluble salts during longer treatment (Xiao & Meng, 2020). Nutrient availability will be elevated with high EC but may also cause a problem of salinity risk if applied to sensitive soils.

Macronutrient Content (N, P, K, Ca, Mg), and Cation Exchange Capacity (CEC)

All the measured macronutrients were significantly increased with the processing time, which shows that the mineral matter is conserved or concentrated because volatile losses are minimised under the specified conditions. Nitrogen (N) increased 0.93 to 1.08, which indicates that the mineral matter is concentrated as organic matter is reduced at any rate. Phosphorus (P) and Potassium (K) also showed increased trends, and they can be explained by the conservation of mineral matter, as volatile losses are minimised in the given conditions. Calcium (Ca) and Magnesium (Mg) rose, as well, which supports the forecast of enriching ash and forming stable mineral phases based on the attractive effect

Table 3
Mean (± s.e.) effects of different residence time (60, 120, and 180 minutes) on the hydrochar chemical properties

Temp.	Time (Mins)	pH	EC (mS/cm)	N (%)	P (%)	K (%)	Ca (%)	Mg (%)	CEC $Cmol_+/kg$
240 °C	60	8.63±0.028^c	1.64±0.006^c	0.93±0.011^b	0.78±0.006^b	1.14±0.006^c	13.31±0.028^c	1.40±0.006^b	7.06±0.009^c
	120	8.72±0.380^b	2.06±0.006^b	0.96±0.018^b	0.83±0.012^b	1.34±0.038^b	14.40±0.035^b	1.47±0.009^a	7.56±0.003^b
	180	8.77±0.028^a	2.69±0.006^a	1.08±0.006^a	0.87±0.009^a	1.45±0.012^a	14.54±0.038^a	1.50±0.015^a	7.91±0.012^a

Note. Means with the same letter in the same column are not significantly different by the Duncan multiple range test at $p \leq 0.05$

of thermal treatment, whereby the disappearance of volatile organic matter causes the relative enrichment of non-volatile nutrients in the solid product. N, P, K, Ca, and Mg are quite high, which confirms that it can be used as a slow-release mineral supplement. It is unlikely that the nutrients will be in the unstable forms, and the chances of leaching are reduced compared to synthetic fertilisers (Joseph et al., 2021).

The cation exchange capacity (CEC) of poultry litter-eggshell hydrochar was determined and presented in Table 3. There was a significant increase in CEC with increasing HTC residence time at the processing temperature of 240 °C. The CEC values increased from 6.95 in the raw feedstock to 7.06, 7.56, and 7.91 cmol(+) kg^{-1} after 60, 120 and 180 min of HTC treatment, respectively. It noticed about 13% increase in the CEC after 180 mins indicating that longer HTC improved the ion-retention power of the produced hydrochar by enhancing the availability of exchange sites, increasing the reorganisation of the mineral and facilitating structural redistribution with the carbon matrix. However, prolonged HTC residence time conditions cause the oxygen-containing functional group to be lost due to the dehydration and decarboxylation process. Integration of eggshells as a Ca-rich mineral with poultry litter improved the CEC. The increase in the associated minerals at the exchange sites and strengthened organo-mineral interaction within the hydrochar could be the result of these mineral components. Previous research has shown that HTC affects the properties of poultry-litter-derived hydrochars by modifying nutrient retention, mineral transformation and surface characteristics (Ghanim et al., 2018; Mau et al., 2016). Similarly, carbon stability and mineral content may be enhanced through HTC-induced modifications in manure-derived hydrochars, which can subsequently influence surface charge characteristics (Li et al., 2022). Similar results were reported by Zhang et al. (2015), where CEC increased in hydrochar produced at 260 °C for 1, 6, and 12 hrs respectively. The relatively small 4.63% increase in CEC was observed between 120 and 180 min, showing that the hydrochar was reaching a more stable state, contributing to lower benefits from future increases in residence time. Overall, these results confirm that HTC effectively improved the functional properties of poultry litter-eggshell hydrochar, enhancing its suitability as a nutrient-retaining soil amendment and a potential material for environmental remediation applications.

Nutrient Recovery

The nitrogen (N) content of the hydrochar produced at 240 °C for 180 mins was higher than that of the hydrochars produced at 60 and 120 mins. Additionally, nitrogen recovery from the hydrochar at 180 mins residence time was significantly higher (Figure 2a). Surprisingly, the hydrochar at 180 mins preserved 43.75% of the initial N, compared with lower recovery rates of 38.85% and 37.65% for 120- and 60-mins residence times, respectively. These results confirmed that longer residence time improves N retention during

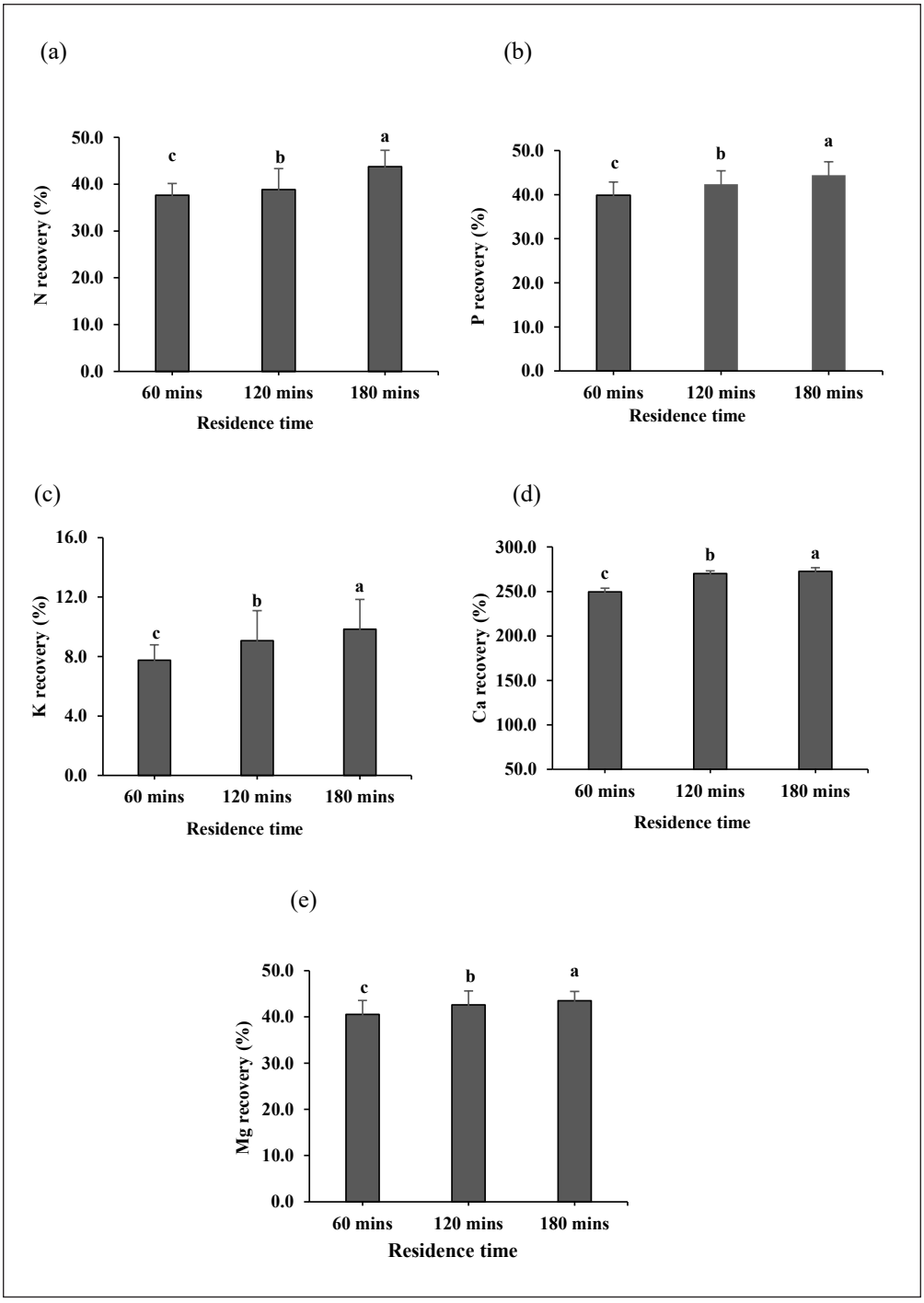

Figure 2. (a) Nitrogen (N), (b) phosphorus (P), (c) potassium (K), Calcium (Ca), and Magnesium (Mg) recovery rates in percentage for hydrochar from poultry litter and eggshell. Different letters above the bars denote statistically significant differences in nutrient recovery among the treatments

the hydrothermal carbonisation process. The increase in nitrogen concentration of the hydrochar with prolonged residence time could be as the result of progressive hydrothermal transformation of the feedstock and nitrogen redistribution carbonisation. As residence time increases, more reactions such as hydrolysis, dehydration and decarboxylation occur, improving the removal of large carbon and oxygen-containing components into liquid and gaseous phases. Therefore, the decrease in the mass of the solid part can lead to a relative enrichment of nitrogen in the remaining hydrochar. Furthermore, increasing the hydrothermal residence time promotes more condensation and polymerisation reactions between nitrogen-containing compounds and carbohydrate-derived intermediates, thereby incorporating nitrogen into more stable solid structures. Longer residence times can also promote the formation and condensation of N-containing heterocyclic structures, thereby increasing the retention of nitrogen in the solid hydrochar (Leng et al., 2021).

In contrast, the recovery rates of P, K, and Mg for hydrochar produced at 240 °C for 60, 120, and 180 mins residence times were slightly below 100%, indicating that most of these nutrients were lost during the production process (Figures 2b, c, and e). Conversely, the Ca recovery rates for different residence times slightly exceeded 100%, indicating that virtually no Ca was lost during the production process (Figure 2d). Residence times of 180, 120, and 60 mins recovered about 272.80, 270.20, and 249.72 of Ca, respectively. It was generally observed from the recovery data that even if the prolonged residence time preserves most of N, P, K, and Mg (>60%), they could conserve almost 100% of the Ca in the poultry litter and eggshell.

X-ray Diffraction Analysis

The X-ray diffraction (XRD) patterns of the poultry litter and eggshell hydrochar produced at 240 °C for 60, 120, and 180 mins residence times were presented in Figure 3 and found to be complex, indicating various peak patterns with some degree of order in short-range, showing an amorphous material. Such characteristics made it difficult to attribute minor peaks to specific minerals, corroborating Da Silva Mendes et al. (2021). Irrespective of the different carbonisation residence times, the hydrochar showed similar peaks and had a wide range of amorphous hump on them, which showed that it had a lot of organic content and little crystalline minerals. There were only a few spectral patterns that had to do with quartz, sylvite, or small amounts of phosphates. The poultry litter-eggshell hydrochar produced showed a different and intense pattern of peaks characteristic of crystalline calcite ($CaCO_3$), particularly the dominant reflection at approximately 29-30° 2θ, alongside several secondary Ca-based mineral peaks. This confirms that eggshell-derived $CaCO_3$ remained stable under hydrothermal conditions and became uniformly incorporated into the hydrochar matrix. The presence of high calcite crystallinity indicates strong liming potential, as $CaCO_3$ showed a high impact for neutralising soil acidity and reducing exchangeable aluminium

in acid soils. The high abundance of calcite at 29-30° 2θ was aligned with the finding of Yang et al. (2023) from the hydrochar produced with sawdust and calcined eggshell.

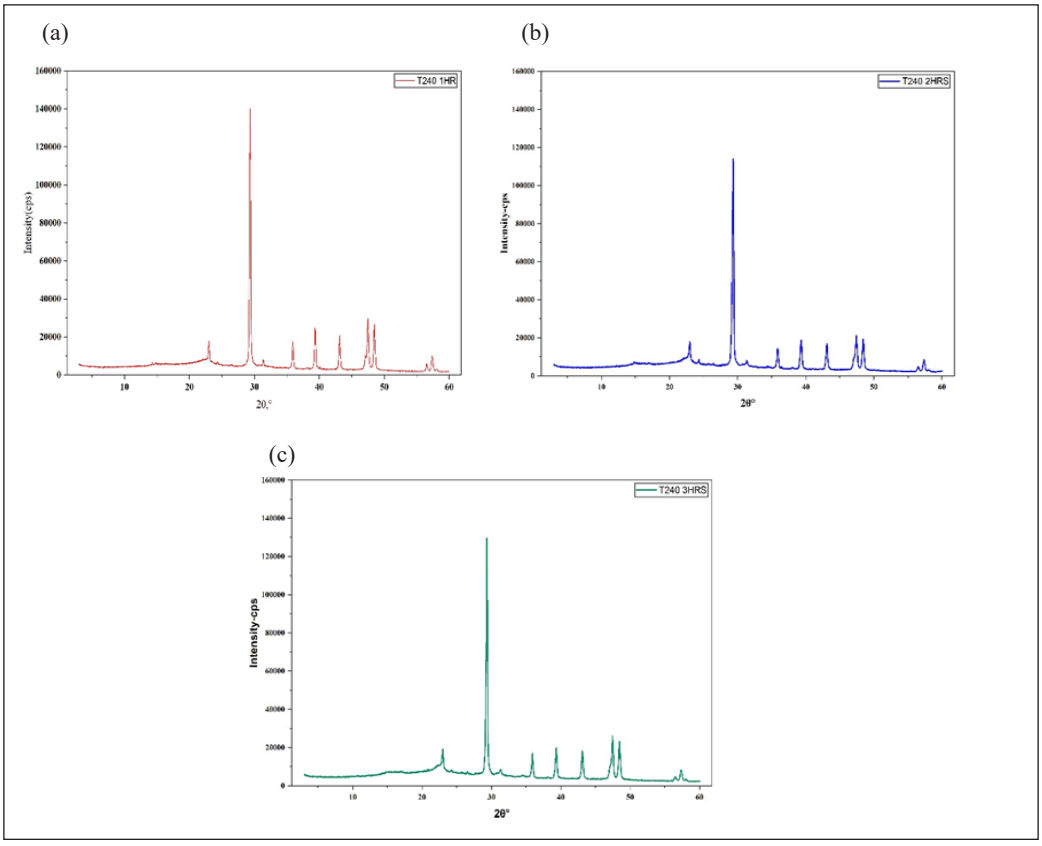

Figure 3. XRD analysis images of a, b, and c poultry litter and eggshell powder hydrochar at 240 °C for 60, 120, and 180 mins, respectively

Morphology Transformations

The colour of the resulting hydrochars was observed based on qualitative visual inspection of their physical appearance. No instrumental colour measurement was performed. The original appearance of poultry litter was a brown powder Figure 4. Under the processing temperature of 240°C at different residence times of 60, 120, and 180 mins respectively, physical changes were observed in the appearance of the hydrochar. The heterogeneous appearance with observed light brown PL particles and pale off-white eggshell fraction was exhibited by the hydrochar at 60 mins residence time. A coarse and particulate surface, related to little thermal activity and minimal structural transformation, was also seen. At 180 mins residence time, the materials became darker, indicating the appearance of organic matter degradation; more homogenised materials were also noticed, and the

eggshell particles became less distinguishable, and the surface texture shows more softened and smoother. The hydrochar looks darker and more uniform, probably because of high thermal decomposition. Eggshell particles are fully integrated into the matrix, and the surface is smoother with finer particles, indicating chemical transformation with longer residence time. The colour changes were the result of dehydration, decarboxylation, and carbonisation reactions occurring during the hydrothermal treatment process. Prolonged residence times also caused the hydrochar to change from light brown to dark brown or black. However, temperature had a more significant effect on the color development than residence time (Yao et al., 2016)

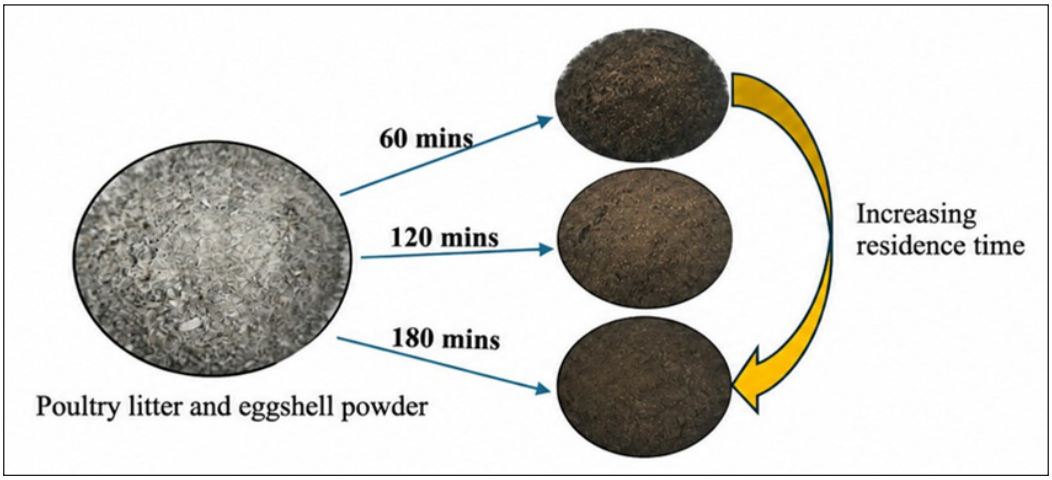

Figure 4. Hydrochars macroscopic morphologies under different residence times

Scanning Electron Microscopy (SEM)

The Scanning electron microscopy (SEM) of PL and ES powder hydrochar produced at 240 °C carbonisation temperature under 60, 120, 180 mins residence time are presented in Figure 5 and reveal a significant difference in their structure and composition, which are highly important for their use. A clear morphological transformation was observed, and it had a distinct morphology, characterised by mineral-rich, granular, and less porous surfaces under different residence times. At a residence time of 60 min, the hydrochar exhibited relatively few pore spaces, suggesting limited structural degradation and greater retention of the feedstock's original mineral constituents, including calcium carbonate derived from the eggshells (Clarke & Olea, 2024; Yang et al., 2023). Hydrothermal carbonisation of PL mixed with ES resulted in the introduction of numerous sphere-like particles in the hydrochar. These sphere-like particles presented a morphology like that of hydrochar porous carbon derived from sucrose and glucose (Ischia et al., 2023; Sevilla et al., 2021). This could be due to the breakdown of cellulose and hemicellulose into glucose during

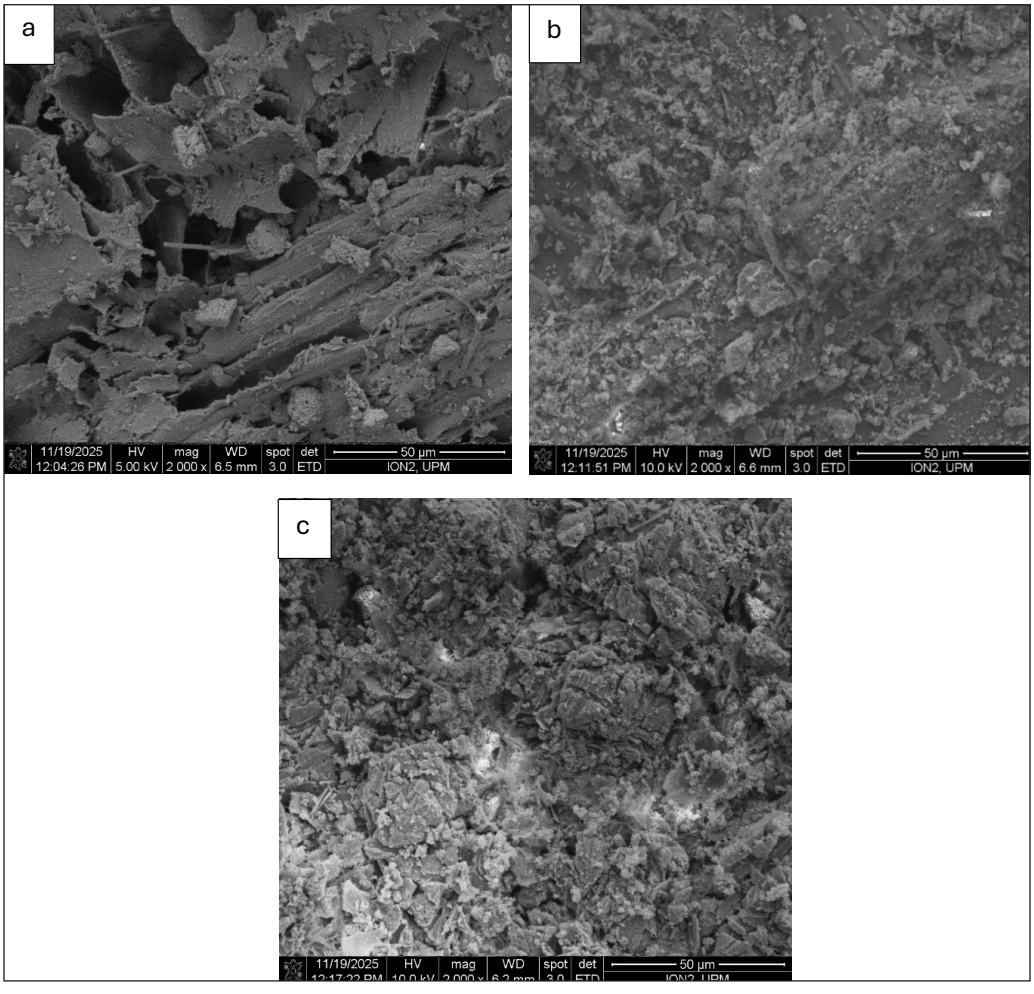

Figure 5. SEM images of a, b, and c poultry litter and eggshell powder hydrochar at 240 °C for 60, 120, and 180 mins, respectively

hydrothermal carbonisation, which enhances the formation of such spherical structures (Cheng et al., 2025).

The carbon in the hydrochar looks like irregular, dense clusters with minimal visible pore space. This is because it is usually processed at a low temperature of 240 °C, which doesn't get rid of enough volatile components that are needed for higher porosity. Clarke and Olea (2024) reported analogous dense hydrochar formations were identified in poultry manure and several nutrient-dense feedstocks treated at temperatures below 260 °C. Several brilliant, angular particles were seen in the hydrochar matrix. These particles are found to be minerals comes from ES, like calcium carbonate and other calcium compounds. Low-temperature hydrothermal processing keeps these minerals intact and evenly spread out

(Shrestha et al., 2024). Their presence offers hydrochar a significant ability to neutralise pH, helping reduce exchangeable aluminium by displacing cations (Lima et al., 2024), and lets the substance work as both an organic carbon source and a mineral liming agent. This is in line with research that shows that hydrochars that have been treated with ES work better than those that haven't (Liu et al., 2022; Shrestha, et al., 2024). Studies show that mineral exposure, not porosity, is what makes low-temperature hydrochars so good at correcting acidic soils (Liu et al., 2025; Shrestha et al., 2024).

Fourier Transform Infrared Spectroscopy (FTIR)

The FTIR spectra Figure 6 of the poultry litter and eggshell powder hydrochar showed a broad and strong band at ~3300 cm^{-1}, corresponding to O-H stretching, which gradually decreased in intensity as HTC residence time increased, indicating gradual dehydration (Islam et al., 2021; Khan et al., 2022) The high peaks at ~1650 and 1540 cm^{-1} (Amide I and II) from poultry litter proteins decreased considerably over 2 hours; hence, confirming the thermal decay of labile nitrogenous substances (Sevilla & Fuertes, 2009). It is worth noting that the typical doublet of calcite in eggshells at 1450 and 875 cm^{-1} did not show in all treatment times, which proves the integration of stable calcium carbonate into the hydrochar structure (Chen et al., 2019). The relative proportionate rise in the salience of these mineral peaks at 180 mins would imply a concentration effect through the loss of organic matter, hence creating a mineral-enhancing carbon resource.

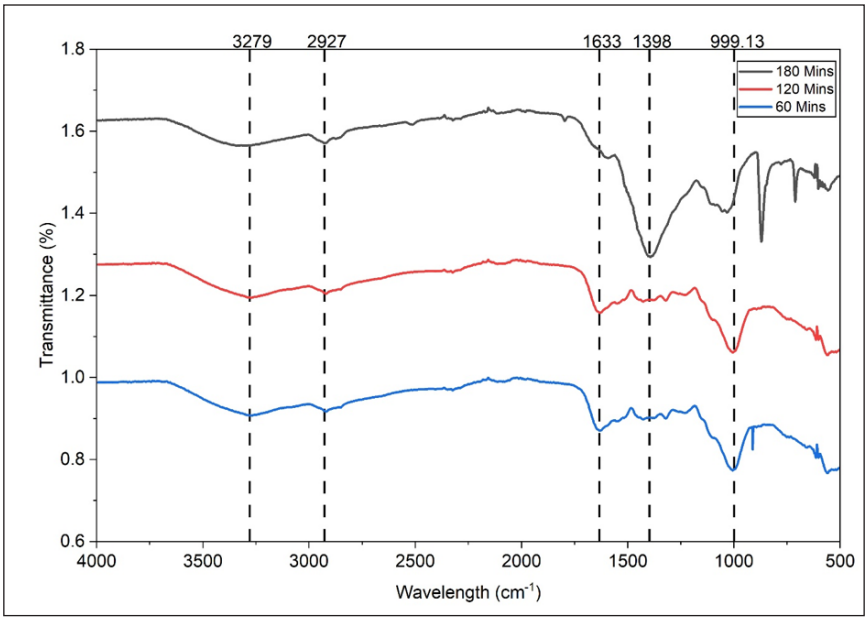

Figure 6. FT-Infrared spectra of poultry litter and hydrochar produced using hydrothermal carbonisation at 240 °C for 60, 120, and 180 mins, respectively

CONCLUSION

The study confirmed that production of hydrochar using poultry litter (PL) and eggshell (ES) waste through hydrothermal carbonisation at 240 °C acquires auspicious quality for use as a soil amendment. Porous structures, increased carbon content, reduced H/C and O/C ratios, and the presence of hydroxyl and carboxylic functional groups, showing improved carbonisation and good surface properties, were observed from the hydrochars. The embodiment of eggshell-derived calcite gave the alkaline nature of the hydrochar, making it suitable for improving soil acidity. Among the various conditions determined, 180 mins residence time gave hydrochar with the most suitable physicochemical properties. Generally, the results highlighted the potential of PL and ES-produced hydrochar as a sustainable and environmentally friendly soil amendment for improving soil fertility, increasing nutrient retention, and correcting soil acidity while encouraging the beneficial reuse of agricultural waste. It is recommended that future studies be carried out to evaluate crop performance from the produced hydrochar through controlled or field trials to determine its effectiveness in improving soil physicochemical properties, nutrient availability, and crop growth and yield, especially on acidic soil.

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