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RESEARCH ARTICLE

SYNERGETIC RELATIONSHIP BETWEEN HYDROPHOBICITY AND BULK DENSITY OF TORREFIED MANGO SEED KERNEL

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ABSTRACT

This study investigated the synergetic relationship between hydrophobicity and bulk density of torrefied mango seed kernel (MSK) as a low-cost biosorbent for water and wastewater treatment. MSK was torrefied at 200 to 300°C and characterized for mass loss, bulk density, and hydrophobicity using standard methods. Hydrophobicity was determined using the water drop penetration time (WDPT) test. Also surface functional groups of untorrefied and torrefied MSK were characterized using Fourier Transform Infrared Spectroscopy (FTIR). Results showed that mass loss increased linearly with torrefaction temperature, from minimal losses at 200 to 225°C (3.33–4.67%) to substantial losses at 250 to 300°C (48.67 to 62.33%), indicating progressive devolatilization and carbonization. Bulk density exhibited a segmented response, increasing at low torrefaction temperatures (≤ 225 °C) due to limited degradation, but decreasing markedly at higher temperatures (250 to 300°C) with a strong inverse correlation to temperature ($r = -0.91$), attributed to pore development and structural breakdown. All torrefied samples were classified as extremely hydrophobic, with WDPT values ranging from 25,880 to 34,105 seconds. Hydrophobicity showed stronger sensitivity to torrefaction temperature than bulk density. FTIR spectra confirmed the progressive loss of oxygen-containing functional groups (O–H, C=O, and C–O) with increasing temperature, explaining the enhanced hydrophobicity. Overall, torrefaction produced a stable, highly hydrophobic MSK biosorbent with reduced bulk density at higher temperatures, highlighting hydrophobicity as a more critical parameter than bulk density in optimizing torrefaction conditions for sustainable biosorbent activation.

KEYWORDS

Biomass, Biosorbent, Sustainable Manufacturing, Torrefaction, Water treatment

1. INTRODUCTION

With the emergence of recalcitrant pollutants, more stringent national and international guidelines on drinking water and discharge effluent quality, and the need for sustainable manufacturing, biosorption has emerged as a promising alternative to the traditional adsorption process. Biosorption involves the use of biosorbents obtained from biomass. Agricultural waste is often the most used biosorbent because of its availability, relatively low cost, and environmental friendliness (Ungureanu et al., 2023; Asadu et al., 2021). However, in their natural state, biosorbents often exhibit low adsorption capacity, prompting the need for modification of their physical and chemical properties (Lima and Ascencios, 2021).

Common approaches to biomass modification include physical activation by carbonization, which involves heating biomass above 350°C in an inert atmosphere, and chemical activation, using reagents such as acids or bases (Ngah and Hanifiah, 2008). Effective carbonization is energy-intensive and environmentally unfriendly, motivating the exploration of low-temperature alternatives to minimize environmental impact (Omoruwou et al., 2022). Torrefaction, conducted at 200-300°C under inert conditions, improves the physical and chemical properties of biomass by reducing moisture and volatiles, increasing hydrophobicity and porosity (Tumuluru et al., 2021; Dyjakon et al., 2022).

Hydrophobicity and bulk density are key physical factors in assessing adsorbent quality. Bulk density impacts how well an adsorbent is packed and is inversely related to porosity. Porosity improves surface area and exposes more active sites (functional surface groups), thereby increasing the adsorptive capacity of the sorbent (Elgarachy et al., 2021). On the other hand, surface hydrophobicity significantly impacts the adsorption of hydrophobic organic pollutants through hydrophobic interactions and water repellency. As reported the enhanced adsorption capacity of modified sphagnum moss biosorbent with improved hydrophobicity for motor oil (Ren et al., 2025).

Although torrefaction has been widely applied to enhance biomass fuels, few studies have investigated its potential as a biosorbent modification technique (Lee et al., 2021; Lu et al., 2021; Kekik et al., 2022; Dyjakon et al., 2022). Most biomass investigated has used seeds of exotic and non-exotic fruits, but none have investigated the effect of torrefaction on seed kernels, especially the mango seed kernel (MSK), even though the mango seed kernel has higher reactivity in thermal treatment than the mango seed (Dyjakon et al., 2022). Also, the potential synergetic relationship between hydrophobicity and bulk density is scarcely reported. This study examined the effect of torrefaction on the physicochemical properties of mango seed kernel (MSK) for enhanced adsorption in wastewater treatment. The use of torrefied biomass-derived adsorbents in wastewater treatment offers a cost-effective and low-carbon footprint for water treatment methods, as well as encourages a circular economy and boosts environmental stability. This supports several Sustainable Development Goals (SDGs), especially SDGs 6, 12, and 13.

2. MATERIALS AND METHODS

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2.1 Sample Preparation and Modification of MSK

Mango seeds were deshelled, and the kernels were crushed to a particle size of 1mm and oven dried at 105-150°C until the moisture content was below 10%. The torrefaction process was carried out in a muffle furnace preheated at 150°C to remove residual moisture, then sequentially heated at 200°C for 60 minutes. This torrefaction process was repeated at temperatures of 225, 250, 275, and 300°C. After cooling in desiccators, the torrefied samples were ground to 125µm and stored in an airtight container for physicochemical analyses.

2.2 Determination of Physicochemical Properties of MSK

Bulk density and mass loss of MSK were determined using a standard container and gravimetric methods, respectively. The container method employed Equation 1, while the gravimetric method employed Equation 2.

$$\text{Bulk Density } (\rho\text{bulk})(g/cm^3) = \frac{\text{Mass of sample in the vessel (g)}}{\text{Volume of the vessel } (cm^3)} \quad (1)$$

$$\text{Mass Loss (ML)} = \frac{\text{Initial Mass} - \text{Final Mass}}{\text{Initial Mass}} \times 100\% \quad (2)$$

Hydrophobicity of the torrefied and untorrefied samples was determined using the Water Drop Penetration Time (WDPT) Test following the classification shown in Table 1 (Dyjakon et al., 2022; Doerr, 1998). This test was done in triplicate for samples torrefied at 200°C, 225°C, 250°C, 275°C, and 300°C, as well as the raw and dry but not torrefied MSK samples.

Table 1: Classification Criterion of Hydrophobic Properties

Time of the Penetration of a Drop of Water (s)	Hydrophobic Properties
<5	Hydrophilic
5 – 60	Slightly hydrophobic
60 – 600	Strongly hydrophobic
600 – 3600	Severely hydrophobic
>3600	Extremely hydrophobic

Surface functional groups of MSK samples were determined by FTIR analysis using an Agilent Cary 630 FTIR spectrometer (Agilent Technologies, USA). Spectra were collected in the range of 4000-650 cm^{-1}, at a resolution of 8cm^{-1}, with 32 scans for both samples and background. Data acquisition and processing were performed using Agilent MicroLab software. MSK sample identification (ID) is shown in Table 2.

Table 2: Description of Samples

Sample ID	Description of sample
RMS	Raw mango seed kernel
DMS	Dry not torrefied MSK
T200	Torrefied MSK at 200 °C
T225	Torrefied MSK at 225 °C
T250	Torrefied MSK at 250 °C
T275	Torrefied MSK at 275 °C
T300	Torrefied MSK at 300 °C

3. Results and Discussion

3.1 Effect of Torrefaction Temperature on Physicochemical Properties of MSK

3.1.1 Mass Loss and Colour Change of MSK

Mass loss was determined for all samples except RMS and DMS because RMS and DMS do not experience mass loss. This is because devolatilization of volatile gases and hydrocarbons occurs at temperatures above 150°C. Figure 1 shows the effect of torrefaction temperature on the mass loss of the MSK. It was observed that there was a linear relationship between torrefaction temperature and mass loss. Mass loss was minimal at the lower torrefaction temperatures of 200°C and 225°C, and increased significantly at mid to high torrefaction temperatures (250-300°C). This is because at lower and higher torrefaction temperatures, torrefaction removes unbound and bound water, respectively (Jaya et al., 2010). Similar linear relationship between mass loss and temperature was also recorded for corn hub, marula seed, and blue gum wood (Orisaleye et al., 2022, Pahla et al., 2018).

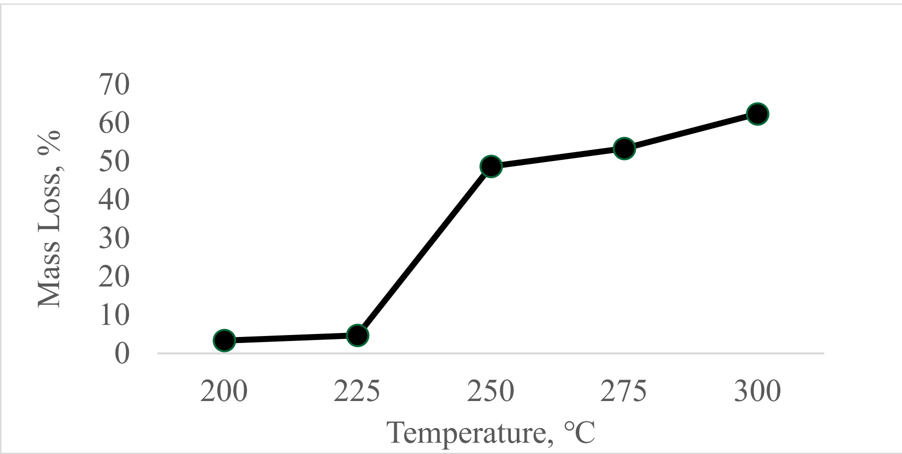

Figure 1: Effect of Torrefaction Temperature on Mass Loss of Mango Seed Kernel Samples

Also, a correlation between mass loss and colour change was observed. At 200-225°C, the minimal mass loss (3.33-4.67%) resulted in minimal colour changes (Figure 2), indicating mild torrefaction characterized mainly by unbound water removal as well as limited devolatilization and carbonization of hemicellulose (Bergman et al., 2005a). At 250°C, a sharp increase in mass loss was observed (48.67%), which indicates the onset of bound water removal and intense thermal degradation associated with hemicellulose and limited decomposition of cellulose (Orisaleye et al., 2022). Decomposition of hemicellulose and cellulose causes biomass to change color due to loss of water, CO_2, and large amounts of acetic acid and phenols (Jaya et al., 2010). Further temperature increases to 275 and 300°C resulted in higher mass losses of 55.33% and 62.33%, respectively, which is an indicator of severe devolatization and the development of a carbon-rich structure marked by a char-like (black) colour. Figure 2 shows the progressive darkening of the MSK samples with increasing process temperature. The colour of the torrefied MSK samples changed progressively from a light brown to an almost black (like char) colour.

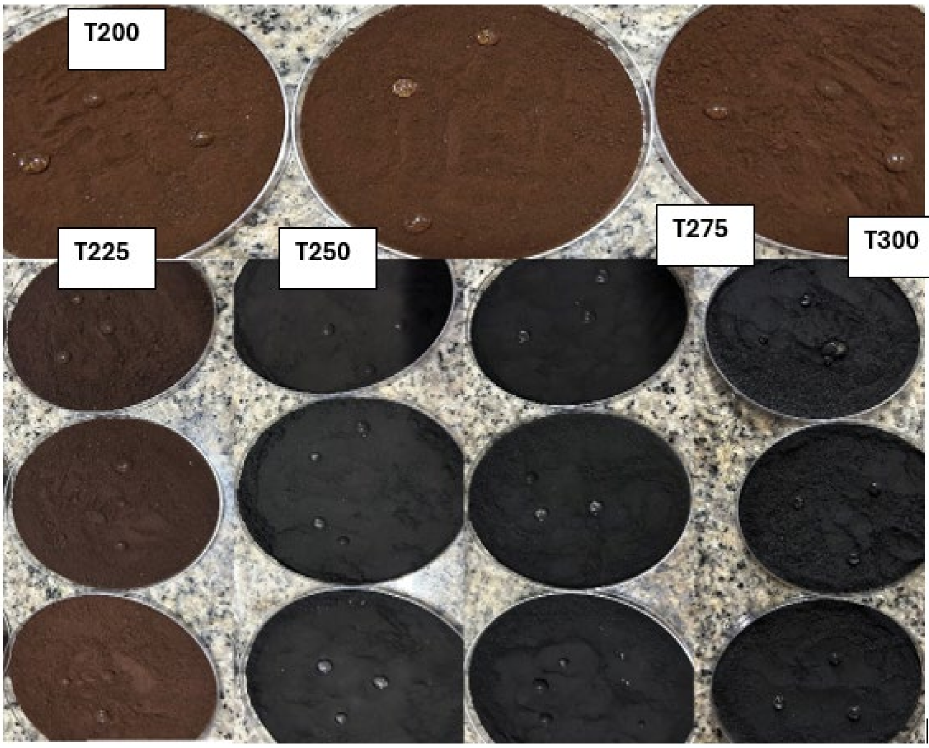

Figure 2: Colour Change in the MSK at Various Torrefaction Temperatures

3.1.2 Bulk Density of MSK

Figure 3 shows the effect of torrefaction temperature on the bulk density of MSK in two segments. A low temperature segment ranging between 200 and 225°C and a mid to high temperature segment ranging between 250 and 300°C. The effect of temperature on the bulk density of MSK was linear at low process temperature and inversely linear at mid to high process temperature, with a correlation value of -0.90. A similar trend was observed in the work of (Dyjakon et al., 2022).

At low process temperature, varying physicochemical changes occur in biomass. According to the study, at 100°C, only drying occurs, keeping hemicellulose and cellulose unaffected, while softening of lignin occurs (Bergman et al., 2005a). Between 150 and 200°C, bound water is lost, cellulose remains unaffected, while hemicellulose and lignin experience depolymerization and recondensation. Between 200 and 250°C, more bound water is lost, and hemicellulose experiences limited devolatilization and carbonization. While lignin and cellulose experience depolymerization and condensation at the lower band, devolatilization and carbonization occur at the higher band. This implies that at low torrefaction temperatures of 200°C and 225°C, a relative increase in the cellulose and lignin content over hemicellulose due to limited devolatilization of cellulose and lignin components would have occurred in the MSK samples, and the resultant effect was the increase in bulk density (Figure 3). With further increase in temperature (250-300°C), all three components experience extensive devolatilization and carbonization in the order of $hemicellulose > lignin > cellulose$ (Bergman et al., 2005a). This infers that as the temperature increased to 250°C, the loss of the hemicellulose, lignin, and cellulose components of the MSK resulted in a steady decrease in bulk density. The initial increase in bulk density with temperature at the low temperature range of 200-225°C explains that within this temperature range, torrefied MSK is relatively unstable and becomes stable with no bound water at the mid to high temperature range of 250 - 300°C.

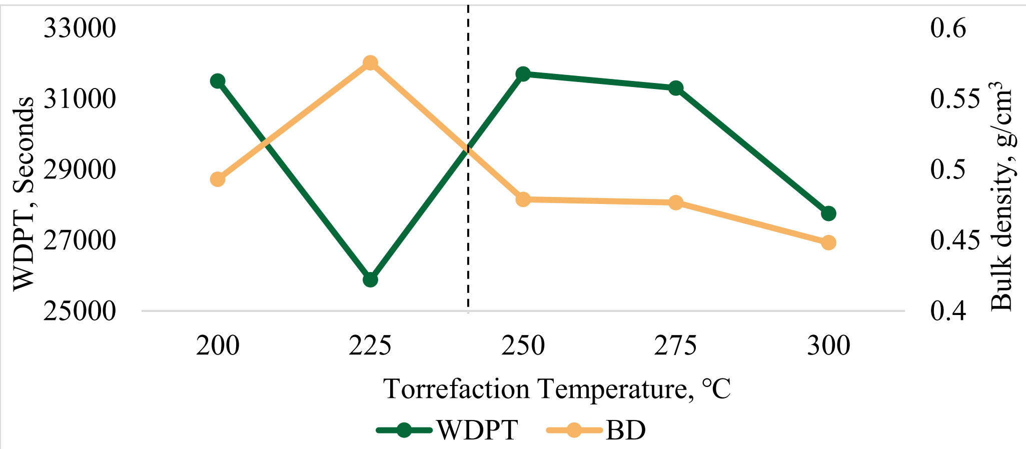

Figure 3: Effect of Torrefaction Temperature on Bulk Density and Hydrophobicity of MSK

3.1.3 Hydrophobicity of MSK

The water drop penetration time (WDPT) values were used to express the hydrophobicity of MSK at varying process temperatures. Figure 3 shows that the WDPT values ranged from 25,880 to 31,700 secs. According to the classification in Table 1, all torrefied MSK samples are extremely hydrophobic (WDPT > 3,600 sec). The generally high WDPT values are consistent with a previous work (Dyjakon et al., 2022). However, the values in this work are higher than those of the previous work and this can be attributed to the varying materials used. While mango seed was used in the previous work, the kernel was used in this work, and the kernel has been reported to have higher reactivity in thermal treatment than the seed (Dyjakon et al., 2022).

Like bulk density, a segmented effect of temperature on hydrophobicity was observed, as shown in Figure 3. A low temperature segment ranging from 200 to 225°C and a mid to high temperature segment ranging between 250 and 300°C. In the low temperature range segment, temperature linked directly with bulk density and inversely with hydrophobicity with 1 and -1 correlation values, respectively. The decrease in hydrophobicity with temperature (from 200 - 225°C) is attributable to the loss of oxygen moieties related to hemicellulose and lignin. However, the sharp increase in hydrophobicity at 250°C supports the extensive devolatilization and carbonization of all three components (hemicellulose, cellulose, and lignin), especially hemicellulose, relatively increasing the cellulose and lignin content over hemicellulose and resulting in the increase in loss of more oxygen-containing moieties. The increased loss of oxygen-moieties may have created new pores resulting in the decrease in bulk density, as shown in Figure 3. According to the study, the formation of pores results in a decrease in bulk density (Azam et al., 2015). The formation of pores also exposes more functional sites that may have decreased the hydrophobicity at higher process temperatures of 275°C and 300°C (Figure 3). This is in agreement with the direct relationship between porosity and surface area.

3.1.4 MSK Surface Functional Groups

Figure 4 reveals FTIR spectra of raw and thermally treated MSK samples segmented into low and high process temperature spectra. The low temperature spectra include spectra of raw (RMS), dry but not torrefied (DMS), and torrefied at 200°C and 225°C temperatures (T200 and T225) while the high temperature spectra include spectra of samples torrefied at 250, 275, and 300°C (T250, T275, and T300) temperatures.

The low temperature spectra reveal the presence of oxygen-containing moieties, including the O–H stretching vibrations of hydroxyl functional groups in the region of 3200–3600 cm^{-1}, the C=O stretching vibrations of carbonyl-containing functional groups appearing in the region of 1650–1750 cm^{-1}, and the C–O stretching vibrations between 1000 and 1200 cm^{-1}. However, the C=O carbonyl group was observed to be distorted with torrefaction temperature. The degree of distortion relates to increasing temperature within the low temperature range. The C–O stretching vibrations appear strong for RMS, T200, and T225 samples but weak in the DMS sample. Another important functional group that appeared in the raw sample and low-temperature-treated samples is the aliphatic C–H stretching vibration between 2850 and 2950 cm^{-1} region. The visible presence of the O-H and C=O groups confirms the presence of bound water in this temperature range.

The high temperature spectra, on the other hand, reveal a flattening of the O-H peak to almost an invisible point with increasing temperature from 250°C – 300°C (T250, T275, and T300). Similarly, the C=O peak weakened while the C–O peaks became almost invisible with increased process temperature. The almost invisible peaks of O-H and C-O peaks, with the weak peaks of C=O suggests the loss of bound water in the torrefied MSK. The aliphatic C–H stretching vibration peak between 2850 and 2950 cm^{-1} region gradually weakens with temperature rise.

The progressive decomposition of oxygen-containing moieties with torrefaction temperature in the high process temperature segment supports extensive devolatilization and carbonization of hemicellulose, cellulose, and lignin, resulting in the characteristic hydrophobicity and bulk density of the torrefied MSK (Figure 4). While the weakening of the C–H peak indicates thermal cleavage of aliphatic chains associated with cellulose, hemicellulose, and lignin components, resulting in increased aromatization and structural re-organization of the torrefied MSK. The FTIR spectra obtained in this study, as shown in Figure 4, are similar to those reported by (Dyjakon et al., 2022).

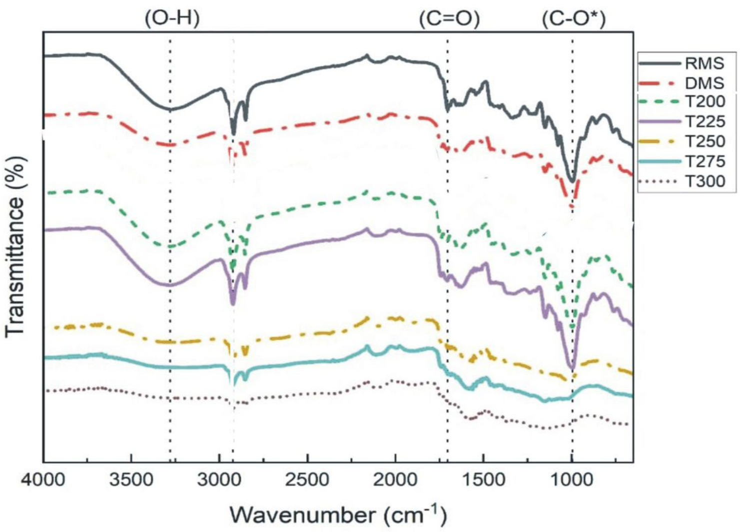

Figure 4: FTIR Spectra of the Mango Seed Kernel at Different Modification Stages

3.2 Relating Physical and Chemical Properties of Torrefied MSK with Hydrophobicity

Figure 3 shows a segmented correlation between hydrophobicity and bulk density with process temperature similar to Figure 4 (FTIR spectra). At low process temperature, an inverse relationship was observed between WDPT and bulk density, while at high process temperature, a direct linear relationship was observed. As earlier stated, this is due to the difference in degradation temperature for hemicellulose, cellulose, and lignin components of MSK. Although the devolatilization temperature for hemicellulose has been reported as 200-250°C, from the FTIR spectra (Figure 4), it is obvious that the hemicellulose of MSK degrades at a temperature greater than 225°C (Bergman et al., 2005a). The loss of hydrophilicity for MSK at low process temperature, therefore, could not be attributed to the loss of the hydroxyl group but carbonyl group as well as the dominance of =C-H through aromatization (Guo et al., 2018). While for the high process temperature samples (T250, T275, and T300), hydrophobicity is due to loss of O–H, C=O, and C–O functional groups.

4. Conclusion

Torrefaction of the mango seed kernel removed bound water and resulted in the loss of oxygen-containing functional groups through devolatilization and depolymerization of hemicellulose, lignin, and cellulose components of MSK. Loss of oxygen-containing functional groups results in hydrophobicity. All torrefied samples were extremely hydrophobic. The effect of torrefaction temperature on the mango seed kernel was segmented into low torrefaction temperature, ranging between 200°C and 225°C, and mid to high process temperature, ranging between 250°C and 300°C. At low temperature, a relatively unstable biosorbent with relatively higher bulk density and improved hydrophobicity was produced, while high temperature produced a stable biosorbent with lower bulk density and a trade-off in hydrophobicity. The effect of torrefaction temperature was more prominent with hydrophobicity than with bulk density, making hydrophobicity a better selection consideration of torrefaction temperature for biosorbent activation in line with sustainable manufacturing.

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