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Valorization of pressmud waste through hydrothermal treatment for energy conversion and liquid organic fertilizer development

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Abstract

Pressmud, a by-product of the sugar industry, contains significant amounts of potassium (K) and phosphate (P) but remains underutilized, leading to waste management and environmental concerns. This study investigates the potential of hydrothermal treatment (HT) to enhance the solubility of K and P for pressmud while assessing its influence on the energy characteristics of the resulting hydrochar. HT was conducted at 180ºC, 200ºC, and 220ºC under non-catalytic and zeolite-catalyzed conditions. The liquid fraction was analyzed for K and P concentrations using a photometer, while the hydrochar was evaluated for calorific value and proximate composition. The optimum treatment condition was achieved at 220ºC without zeolite, yielding the highest K and P solubilities of 1200 ppm and 1790 ppm, respectively. The presence of zeolite decreased solubility due to its ability to adsorb nutrients. Higher nutrient concentrations were positively correlated with increased calorific value and fixed carbon content, with a maximum value of 2047.56 cal/g. These findings demonstrate that hydrothermal treatment is an effective strategy for nutrient recovery and energy enhancement from pressmud waste, supporting its valorization toward renewable energy production and sustainable fertilizer development.

Keywords: hydrothermal treatment, pressmud, potassium, phosphate, hydrochar

1. Introduction

Pressmud is a solid residue produced from the sugarcane purification process in the sugar industry. As a waste with high moisture content (approximately 70-80%), its management often poses a significant challenge, particularly due to its potential to cause serious environmental impacts [1]. Even though pressmud has organic matter and key nutrients like potassium (K) and phosphate (P), it's still not being used to its full potential. Pressmud is often merely discarded or used as fertilizer with limited processing, which still poses a risk of groundwater contamination due to the leaching of the chemical elements it contains [2]. This highlights the need for a more effective and integrated approach to handling pressmud, which can reduce environmental harm while enhancing its economic value. The chemical compounds in pressmud, like phosphate and potassium (Figure 1), have a lot of potential for farming because they help with photosynthesis, water regulation, and plant growth [2]. But in its raw, hot, and poorly processed state, pressmud isn't very useful. With the right processing technology, though, it can be turned into more valuable products.

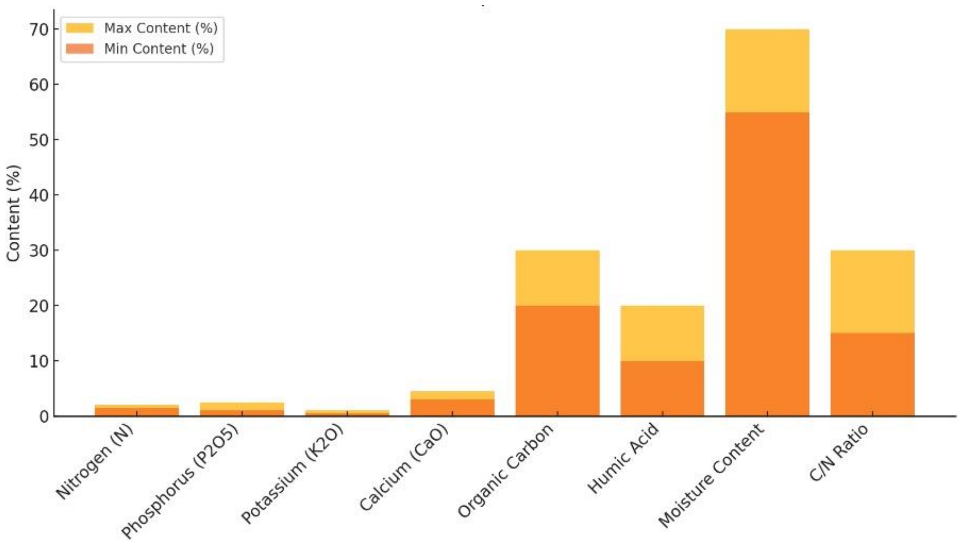


Figure 1. Chemical Compound Content of Pressmud [3]–[5]

One good way to tackle this problem is by using Hydrothermal Treatment (HT). HT breaks down biomass in water at high temperatures and pressure, making it ideal for wet materials like pressmud. It also skips the drying step, which is usually a big hassle with traditional methods [6]. By tweaking the process settings, you can control the amounts of solid, liquid, and gas produced [7]. HT produces two main products: a liquid that can be used as organic fertilizer and a solid called hydrochar, which works well as fuel or a soil enhancer [8].

Previous studies have found that HT helps release key nutrients like potassium and phosphate from different kinds of biomass. But there's still not much research on how HT affects the release of these nutrients from pressmud, or how they influence things like the energy value and overall makeup of the hydrochar.

Things like moisture, ash, volatile matter, fixed carbon, and energy value are important when deciding if biomass can work well as fuel. Changes in phosphate and potassium levels are likely to influence these factors. So, this study isn't just about seeing how HT affects nutrient release from pressmud; it's also about figuring out how these nutrients impact the fuel quality of the hydrochar.

Another important point to look into is the use of a zeolite catalyst during the HT process. Zeolite is known for its strong absorption ability and can help improve how well nutrients are extracted during HT [6], [9]. This study will also check how adding zeolite affects the release of potassium and phosphate, and how it influences the energy quality of the solid product.

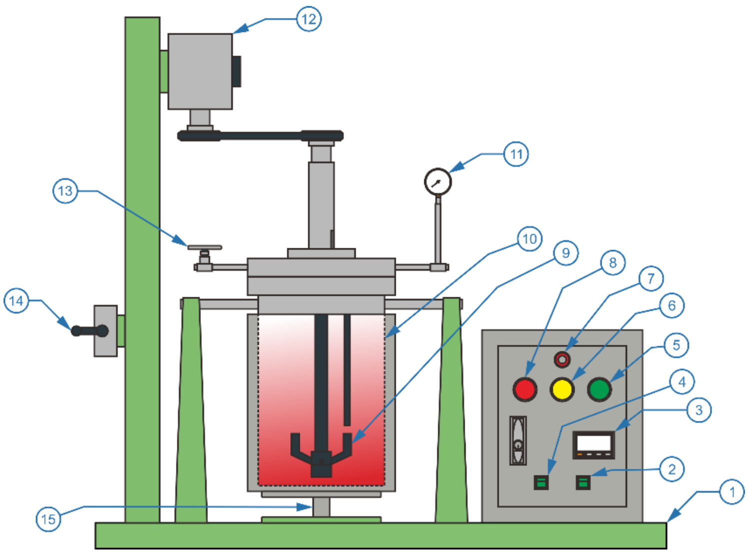
While plenty of research has looked at how HT affects different kinds of biomass waste, there's still not much focus on how potassium and phosphate levels impact the energy value and basic makeup of pressmud after HT. Most past studies have either looked at improving fuel quality or making nutrients more available, but not both together. So far, no study has really dug into how releasing nutrients like potassium and phosphate ties into the fuel quality of hydrochar. Plus, the role of adding zeolite to boost nutrient release and improve the energy quality of the solid product hasn't been explored much either.

This study takes a fresh approach by looking at how potassium and phosphate levels affect both the energy value and basic makeup of hydrochar made through HT. What makes it stand out is the focus on how nutrients relate to fuel quality, something that hasn't been talked about much in previous research. On top of that, using zeolite as a catalyst is another new angle, with the idea that it could help release nutrients more effectively and improve the energy quality of the solid product [6]. This research offers a more well-rounded solution for managing pressmud waste with HT, combining both nutrient recovery and energy benefits to help promote sustainable farming and renewable energy use [8].

2. Methods

A 3 kg sample of pressmud waste with 69% moisture was taken straight from the conveyor at PTPN XI (Persero) PG-Semboro in Jember Regency and stored in an airtight container before freezing. For the experiment, 12 litres of distilled water were sourced from *Alfa Kimia*, Yogyakarta, and 500 g of natural zeolite, sized -4+5 mesh, came from *Rumah Inovasi Daur Ulang* at the Agro Technology Innovation Center, Universitas Gadjah Mada (UGM). The zeolite was heated at 105ºC for five hours before being used.

The hydrothermal reactor, made of stainless steel with a capacity of 2 kg, is equipped with a stirrer, pressure gauge, and temperature controller that automatically adjusts the pressure according to the set temperature (Figure 2). HT was carried out at temperatures of 180, 200, and 220ºC, with a raw material-to-water ratio of 1:5, with and without zeolite, at a stirring speed of 1360 rpm (Figure 3). The volume of zeolite added was 20% of the solid weight.



Details:

1. Chassis
2. On/off switch
3. Temperature controller
4. Heater switch
5. Heater indicator
6. Motor indicator
7. Alarm
8. Power indicator
9. Stirrer
10. Reactor
11. Pressure gauge
12. Motor
13. Pressure valve
14. Motor switch
15. Reactor base

Figure 2. Hydrothermal Reactor Scheme [10]

This study consists of three stages: raw material preparation, the hydrothermal treatment process to examine the effect of temperature and the addition of a zeolite catalyst on the characteristics of the liquid product, specifically focusing on the potassium and phosphate content produced, and investigating their relationship with calorific value, moisture content, ash content, volatile matter, and fixed carbon content, followed by the analysis of results and conclusions. The liquid product analysis aims to identify several organic compounds dissolved in the liquid produced from the hydrothermal treatment. This analysis includes pH, potassium, and phosphate content. The liquid product analysis was conducted at Rumah Inovasi Daur Ulang, Pusat Inovasi Agro Teknologi UGM, using a photometer (Hanna).

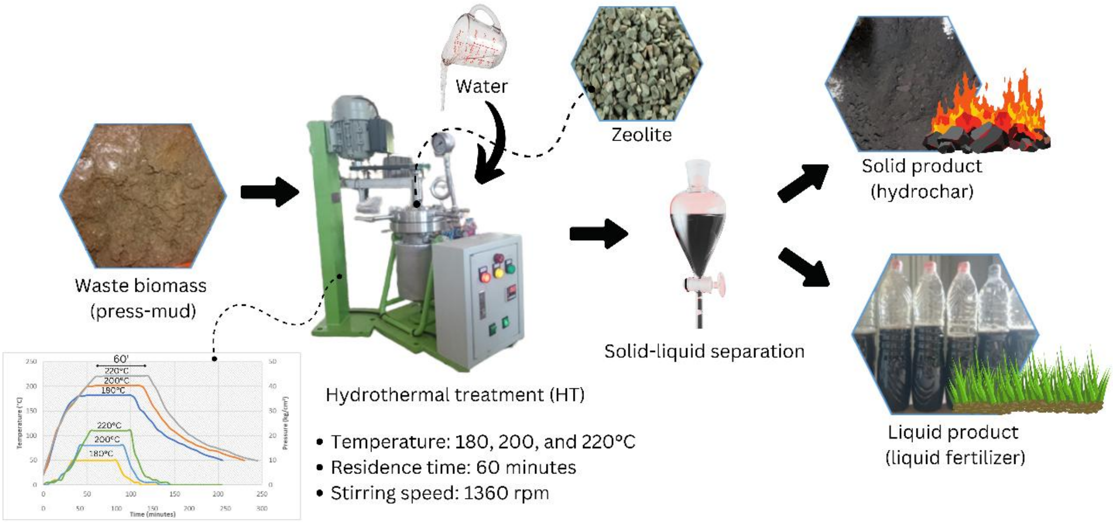


Figure 3. Research Process Scheme

To check the potassium levels, 10 ml of the liquid sample was put into a cuvette, followed by six drops of HI 93750A-0 reagent. After that, the cuvette was placed in the photometer, the device was turned on, and the "zero" button was pressed until it showed "-0.0-," meaning it was ready. Then, HI 93750B-0 reagent was added to the sample, and it was put back into the photometer. The "read" button was pressed, and the result popped up. Phosphate levels were measured the same way, except the HI 93717 reagent was used instead.

3. Result and Discussion

Liquid Product Analysis and the Effect of Temperature

The purpose of analyzing the liquid product is to measure how much inorganic material has dissolved in the water. Elements like calcium (Ca), magnesium (Mg), sulfur (S), phosphorus (P), and potassium (K) from the biomass can dissolve into the water during the hydrothermal treatment process [11]. Studies have looked into how potassium compounds like potassium chloride (KCl) and potassium nitrate (KNO_3) dissolve in supercritical water, showing that potassium does indeed dissolve under hydrothermal conditions [12]. Additionally, potassium is found to be largely dissolved in process water during the hydrothermal processing of cow manure [13]. Liquid product analysis includes potassium levels, phosphate, and pH. Potassium and phosphate are crucial parameters in fertilizer production. The results of the liquid product analysis are shown in Table 1.

Table 1. Results of Liquid Product Analysis from Hydrothermal Treatment

Parameter	Potassium (ppm)	Phosphate (ppm)	pH
HT-180	900	850	4.8
HT-200	1000	1370	4.2
HT-220	1200	1790	4.2
HTZ-180	800	830	4.5
HTZ-200	920	1300	4.1
HTZ-220	1120	1690	4

One of the advantages of hydrothermal treatment in processing pressmud waste is that it can be directly applied to sugar factory waste without the need for a drying process, thereby avoiding becoming a new source of pollution for the environment. The fertilizer production process still uses conventional drying methods, making wet pressmud waste a new problem for sugar factories and surrounding communities due to the odour it produces. Using the hydrothermal treatment process, the pressmud waste is degraded using thermal processes, resulting in no foul odour and being more environmentally friendly.

Table 1 shows that the highest solubility level of potassium is achieved through the hydrothermal treatment process without zeolite at a temperature of 220°C. Potassium is a compound that easily dissolves in water [14]. In various studies, higher temperatures generally increase the extraction or the content of potassium [15]–[19]. The higher the temperature used, the greater the potassium content produced. This is due to the higher temperature in the reactor, which affects the rate of movement and collisions between molecules, leading to a better reaction and greater potassium conversion. This aligns with previous research stating that high temperatures in thermal processes increase the solubility of solvents (water) and can enlarge the pores of solids, allowing the solvent to dissolve solid components [20]. The same phenomenon occurs with phosphate solubility; the higher the temperature, the greater the dissolved phosphate. Higher temperatures during hydrothermal treatment generally enhance the solubilization and extraction efficiency of various compounds, including phosphate, supporting the statement that high temperatures combined with stirring are more effective than lower temperatures in facilitating this process [21]–[25]. Therefore, it can be concluded that high temperature, accompanied by stirring, has a more favourable effect on hydrothermal treatment extraction than lower temperatures. Temperature is a crucial factor in the hydrothermal treatment process. Temperature variations can alter the characteristics and structure of the resulting solid and liquid products.

Physically, the hydrochar produced becomes finer and more homogeneous as the temperature increases. The hydrolysis process will break down the biomass structure into carbon. Hydrolysis is one of the biomass decomposition reactions that uses water as a separator. Hydrolysis reactions occur at temperatures above 180°C and are within the subcritical point of water, which occurs at temperatures below 374°C with pressures less than 22.1 MPa [26]. The hydrolysis reaction begins with the formation of hydronium acid ions (H_3O^+) and hydroxide base ions (OH^-) from the ionization of water as a reactant. The higher the process temperature, the more ions dissolve in the water. A high ionization constant allows subcritical water to provide an acidic medium in hydrolysis [27]–[29].

The Effect of Zeolite Use in the Hydrothermal Treatment Process

The addition of zeolite in the hydrothermal treatment process decreases the dissolved potassium and phosphate content. This is most likely due to the zeolite's absorption of potassium and phosphate elements in the liquid. Mordenite zeolite has a pore size of 0.65 x 0.70 nm, while potassium and phosphate ions have smaller ion sizes of 0.133 and 0.238 nm, respectively [30]–[32]. The smaller size of potassium and phosphate ions compared to the zeolite's pore size can facilitate the absorption of these elements.

The potassium and phosphate contained in pressmud waste can be absorbed up to 1200 and 1790 ppm levels. This process occurs during hydrothermal treatment without zeolite at a temperature of 220°C. When used as fertilizer, potassium and phosphate help with photosynthesis, boost early plant growth, make stems stronger, speed up decay time, support cell nucleus formation, and improve plant resistance to diseases [33].

Zeolite creates a more acidic environment compared to hydrothermal treatment, which only uses subcritical water as a catalyst. It allows for better control of acidity, making it more effective for processes that need a stronger acidic setting [34]–[37]. Table 1 shows that as the temperature goes up, the pH drops. There's also a clear pH difference between treatments with and without zeolite. Adding zeolite makes the process more acidic, shown by a bigger drop in pH compared to when no zeolite is used. It proves that the catalytic effect of zeolite improves hydrothermal efficiency and affects the physical properties of the resulting hydrochar.

pH Analysis and FTIR Characterization in Hydrothermal Treatment with Zeolite

During hydrothermal treatment, the deionized water started with a pH of 6.9, which is slightly alkaline. As the temperature went up, the pH dropped, becoming more acidic and reaching 4.2 at 220°C. Interestingly, the pH stayed about the same between 200°C and 220°C. When zeolite was added, the pH dropped a bit more, by around one point. To double-check the results, a test was also done using just deionized water and zeolite without any raw materials. At 200°C, the pH without zeolite was 4.2, but with zeolite, it went down to 4.0. This shows that the raw materials do not really cause much of the pH drop; it is mostly the heat and the zeolite that make the difference.

When zeolite was added at 180°C, the pH dropped by up to 4 points compared to treatment without zeolite. But at higher temperatures like 200°C and 220°C, the drop was only around 1 to 2 points. This happens because zeolite creates a stronger acidic environment than just using subcritical water. As the temperature rises, the acidic sites on the zeolite increase, lowering the Si/Al ratio and making the environment even more acidic [38].

Besides checking the pH, FTIR analysis was also done to see how the structure of zeolite changes during hydrothermal treatment. FTIR is a standard method for looking at functional groups in zeolite and is often used to track changes in OH group intensity, which shows the presence of water molecules and other impurities [39], [40]. The FTIR results showed that after hydrothermal treatment, the O-H groups in the calcined zeolite had lower intensity, meaning some water molecules were released. This drop in intensity suggests the zeolite got rid of more impurities, leading to cleaner, more open pores [41]. The results of the FTIR analysis are shown in Figure 4.

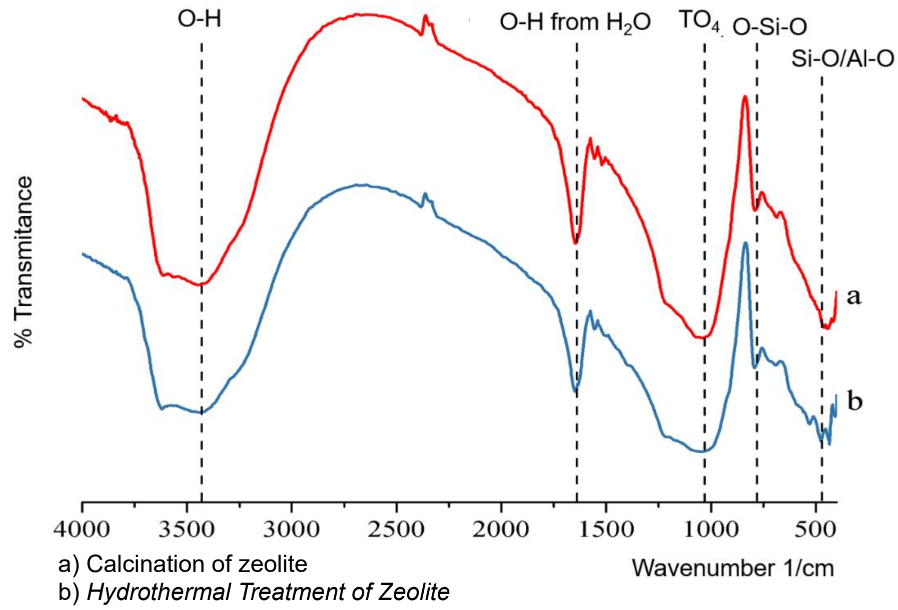


Figure 4. Comparison of FTIR Spectrum of Physically Calcined Natural Zeolite and Hydrothermal Treatment Results

The peak around 1100-1000 cm⁻¹, which relates to the asymmetric Si-O or Al-O groups in TO_4, becomes weaker after hydrothermal treatment. This shows that the Si/Al ratio in the zeolite structure has dropped. Additionally, an increase in intensity at the wavenumber range of 820-650 cm^{-1} indicates structural changes (dealumination) of the zeolite framework [42]. His increase in intensity is also observed in the Si-O/Al-O groups, which become stronger after the hydrothermal treatment. This is further supported by previous research, which states that HT treatment enhances the formation and intensity of Si-O/Al-O bonds [43], [44].

Based on the interpretation of the FTIR results, it can be concluded that hydrothermal treatment reduces the Si/Al ratio in the zeolite, which increases the hydrophilic properties of the zeolite, facilitating water absorption.

The rise in acidity during this process is also linked to the hydration of zeolite crystals, which lines up with earlier pH results showing that adding zeolite lowers the pH.

The Effect of Potassium and Phosphate Content on Calorific Value and Proximate Analysis

In this study, potassium and phosphate levels had a big effect on both the energy value and proximate analysis of the solid products made through hydrothermal treatment (HT) and HT with added zeolite (HTZ). Figure 5 presents the data on calorific value and proximate analysis comes from earlier research, covering heating value, fixed carbon, volatile matter, ash, and moisture levels [10].

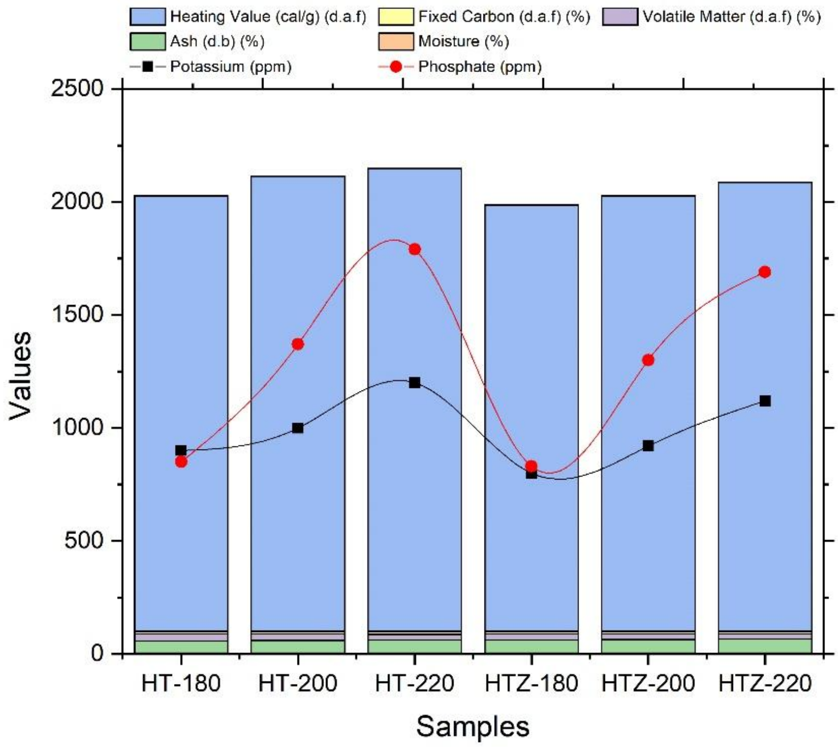


Figure 5. The Effect of Potassium and Phosphate Content on Calorific Value and Proximate Analysis

In the HT-180 sample, where potassium is at 900 ppm and phosphate at 850 ppm with a pH of 4.8, the ash content is 53.853%, volatile matter is 30.984%, and fixed carbon is 11.039%. The energy value comes out to 1926.844 cal/g. As temperatures rise to 200°C and 220°C in the HT-200 and HT-220 samples, potassium and phosphate levels go up to between 1000-1200 ppm and 1370-1790 ppm, while pH drops to 4.2. This jump in potassium and phosphate is linked to an increase in energy value, with HT-200 reaching 2013.917 cal/g and HT-220 topping out at 2047.559 cal/g. Fixed carbon also rises, from 11.039% in HT-180 to 12.514% in HT-220. The higher nutrient levels and temperatures seem to help break down volatile compounds more effectively and strengthen the carbon bonds in the solid product. Basically, the hotter the treatment, the more the biomass breaks down, releasing more ions like potassium and phosphate [45]. Higher temperatures also speed up lignin breakdown and lower the oxidation temperature [46]. At higher temperatures, like in HT-200 and HT-220, the hydrothermal process does a better job breaking down organic materials and encouraging reactions between potassium, phosphate, and other components, which leads to more fixed carbon in the final product.

More fixed carbon leads to a higher calorific value since it’s a main part of the solid that holds energy. On top of that, the extra potassium and phosphate help strengthen the solid structure through chemical bonding within the carbon matrix, boosting the material's energy density. The drop in pH seen in HT-200 and HT-220 shows more acidic activity during the process, which helps break down organic matter faster and makes energy conversion more efficient, as shown by the higher calorific values.

Adding zeolite in the HTZ-180, HTZ-200, and HTZ-220 samples also changes the composition and boosts the calorific value. Although potassium and phosphate levels in the HTZ samples are lower than in HT, ranging from 800-1120 ppm for potassium and 830-1690 ppm for phosphate, the addition of zeolite increases the ash content to 62.467% in HTZ-220. However, the calorific value in HTZ samples tends to be lower than in HT, with HTZ-220 producing a calorific value of 1987.026 cal/g, which is lower than HT-220. Although zeolite can affect the material structure, the increased ash content and reduced volatile matter in HTZ samples reduce the resulting energy density.

pH also plays a role in the results. Samples with lower pH, like HT-200 and HT-220 at pH 4.2, show higher calorific values and fixed carbon compared to samples with higher pH, like HT-180 and HTZ-180. This points to a more acidic environment, helping with better energy conversion and more stable carbon products. Overall, higher potassium and phosphate levels seem to improve the solid fuel quality, with more fixed carbon and a higher energy value. Still, even with high potassium and phosphate, adding zeolite during hydrothermal carbonization tends to increase ash content and slightly lower the calorific value.

4. Conclusion

This study shows that Hydrothermal Treatment (HT) works well for handling pressmud waste without needing to dry it first, which could help cut down on environmental pollution. Running HT at higher temperatures, like 220°C, boosts the release of potassium and phosphate, making it useful for fertilizer. The results also show that adding zeolite lowers the amount of dissolved potassium and phosphate, likely because the zeolite absorbs some of these nutrients. So, HT without zeolite releases more nutrients, while HT with zeolite may help improve extraction efficiency and the energy quality of the solid product.

Overall, this study has implications for the development of liquid organic fertilizers and renewable fuels and the potential to reduce the environmental impact of sugar industry waste. As a next step, further research could explore other catalyst combinations for optimizing the outcomes.

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