Production of Hydrochar from Palm Oil – Based Industrial Sludge by Hydrothermal Carbonization Process

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**Abstract.** Wastewater treatment plants in downstream industries that process crude palm oil (CPO) into products such as food, biodiesel, and oleochemicals generate large amounts of sludge. This sludge, with a high-water content of 43.14% and an unpleasant odor due to residual oil, has not been utilized and poses environmental problems. Therefore, research is being conducted to convert this sludge into a solid fuel with a calorific value comparable to coal, which could serve as a coal substitute. Dry sludge has a calorific value of up to 25.34 MJ/kg, and no harmful metals have been detected in it. Hydrothermal Carbonization (HTC) is employed to enhance the calorific value of the sludge. The HTC process is selected because it condenses the energy content of wet biomass through the application of heat and pressure, eliminating the need for a prior drying step. Compared to combustion and pyrolysis, HTC requires less energy, operates under milder reaction conditions, and produces no secondary pollutants. This study will further investigate the effects of Dry sludge (DS): Water (W) ratios (ranging from 1:3 to 1:11 g/mL), temperature (160-240°C), and reaction time (30-120 minutes). The results indicate that the yield of hydrochar decreases as the DS:W ratio increases. The calorific value increased by up to 7.73% at ratio of 1:5 g/mL but then decreased by up to 3.26% at ratio of 1:11 g/mL. The best energy densification and energy recovery, 1.8 and 76.79% respectively, were achieved at ratio of 1:5 g/mL. Additionally, the yield of hydrochar decreases with increasing reactor temperatures, with a more pronounced decrease (26%) at temperatures below 200°C and a less significant decrease (6.45%) between 200°C and 240°C. Based on the preliminary data, it can be concluded that the HTC process is effective for converting waste sludge into hydrochar, which could be used as a coal substitute. The optimal conditions for the hydrothermal process are a temperature of 220°C, a solid-to-water ratio of 1:5 g/mL, and a reaction time of 120 minutes.

# INTRODUCTION

The palm oil refinery industry is an industry that processes Crude Palm Oil (CPO) to be further processed into food products, biofuels, and oleochemicals. The liquid waste produced by the treatment process will be flowed to the wastewater treatment plant. The liquid waste treatment process produces two final products, namely clear wastewater that flows into water bodies and sludge. Sludge has a foul odor and a relatively high water content (43.14%), due to the amount of oil dissolved in it. The sludge has not been used until now, causing environmental problems. Sludge contains a high calorific value of 25.34 MJ/Kg and does not contain heavy metals (**Table 1),** so it is very good to be converted into hydrochar with a calorific value equivalent to subitumious coal, to be used as a fuel substitute for coal.

Sludge can be converted into hydrochar by using thermochemical processes such as pyrolysis, gasification or combustion. These methods are able to reduce the volume of sludge and turn it into a product that has high value (Pirolisis et al., 2021). Unfortunately, the thermochemical method necessitates drying berforehand, which means costly pretreatment, high energy consumption and generally emitting dioxins and heavy metals. The direct combustion process cannot be carried out because the sludge contains high oil so it will make the sludge stick to the boiler and cause secondary pollution that can harm the environment (Reguler & Du, 2021). The hydrothermal carbonization (HTC) method is the ideal conversion technology for sludge conditions with a high moisture content

**TABLE 1.** Comparison of sludge composition with quality standards for hazardous and toxic waste

|  |  |  |
| --- | --- | --- |
| **Parameter** | **TCLP (mg/L)** | **Sludge Refinery CPO**  **Industry (mg/L)** |
| Arsen, As | 0,5 | <0,001 |
| Cadmium, Cd | 0,15 | <0,06 |
| Boron, B | 25 | <0,10 |
| Lead, Pb | 0,5 | 0,07 |
| Mercury, Hg | 0,05 | <0,002 |
| Nitrat | 2500 | 13,80 |
| Nitrit | 150 | <0,03 |
| Cyanide | 3,5 | <0,03 |
| Cloride | 12.500 | 1,10 |
| Crom, Cr | 2,5 | 0,09 |
| Nickel, Ni | 3,5 | 0,44 |
| Copper, Cu | 10 | 2,2 |

because it applies pressure and heat to compress the sludge’s moist energy content without any prior a pre-drying step. The HTC process operates between 150 – 300 C̊ at pressures between 1.5 Mpa – 5 Mpa, which are higher than saturated pressures remains the water in a liquid state and serveas a catalyst. This matters in thermal processes where biomass moisture is not needed. Preventing energy loss during the drying process of wet raw materials is one strategy to lower the cost of processing biomass with high moisture content. Degradation in the process begins with a hydrolysis reaction followed by dehydration, decarboxylation, condensation, polymerization and aromatization (A. Leena Pauline, 2020). Low oxygen concentrations are preferable to lessen the oxidation of carbon to CO2 and as temperature rises, pressure on the HTC process will grow. Moreover, it will recover more carbon as hydrochar, thus improving combustion efficiency. Water has a subcritical condition dur to high pressure and temperature, which remains it in the liquid phase. This aims to avoid the intensive steps of evaporation energy required in the process to produce hydrochar as well as to avoid the occurrence of explosion during the process. The volume of sludge will also be significantly reduced due to the increase in the dewatering process. Therefore, this method is more effective at turning sludge into hydrochar [1].

Depending on the raw materials and process parameters, the HTC process will convert the sludge into three different distributed product types. These products are generally based on the dry weight of the sample as a base, 50% - 80% into hydrochar (HC), 5% - 20% into the liquid phase and 2% - 5% in the gaseous phase (Zhao et al., 2014). Solid decomposition will be affected by reaction temperature, reaction time and the ratio of solid to liquid to produce yield and determine the characterictics of hydrochar. Reaction temperature is a crucial factor that influences the reaction mechanism and properties of hydrochar, because it provides enough heat to breakdown organic macromolecules and recombine extremely active chemical bonds. Therefore, the hydrothermal reaction will become more active at high temperatures, hastening the rate of sludge components to degrade, decompose, hydrolyze and polymerize. Wang et al (2022) examined the impact of HTC’s process temperature at 150 – 250 C̊ for 30 minutes, and discovered that the hydrochar yield decreased with increasing heating temperature. Hydrochar yield decreased by 11.84% below 190 C̊ and by 4.67% between 190 – 250 C̊ [2]. This phenomenon also occurs during the hydrothermal process of hornwort into hydrochar at 240 – 320 °C for 30 minutes. The result is hydrochar yield decreased by 51.55% at 240 C̊ [3]. Wang et al (2022) investigated the impact of time on the HTC process (30 – 120 minutes) and discovered that, there was a notable contribution to the breakdown of sludge components at a reaction time of 30 minutes. Over the course of the process, The yield dropped by 2.67% for 30 minutes and then by 0.83% for 60 minutes [2]. According to Paiboonudomkarn et al (2022) analysis of the solid-liquid ratio (1:0.25 – 1:3) and found that the ratio of 1:0.5 can be decreasing Volatile Matter and increasing Fixed Carbon [4].

In this study, sludge obtained from the palm oil refinery industry is used to convert into hydrochar as a solid fuel alternative to coal using the hydrothermal carbonization (HTC) method. This study will also study the influence of temperature, reaction time and solid-liquid ratio on hydrochar yield as well as study the fuel properties and chemical characteristics of hydrochar as a solid fuel alternative to coal.

# MATERIAL AND METHODS

## Materials

The raw material was Sludge from Crued Palm Oil Refinery Industry, which was stored in the refrigerator.

Additional components include Distilled water and CO2 gas from Commercial Product.

## Hydrothermal Carbonization Method

A Hydrothermal reactor with an OD of 10.8 cm, an ID of 6 cm, and a height of 10.3 cm, made of Teflon on the inside and Stainless-Steel SS-316 on the outside. The reactor has a volume of 301.05 mL and can function at a pressure below 120 bar and a maximum temperature of 260 °C was used to Hydrochar. In the hydrothermal reactor, 15 g of sludge and 135 ml of water were added. After that, CO2 gas was used to apply a pressure of up to 4 MPa to the reactor's solution. The reactor was then heated for 30 minutes, 45 minutes, 60 minutes, 90 minutes, and 120 minutes, with temperature variations of 160°C, 180°C, 200°C, 220°C, and 240°C. The solid-liquid ratio was studied at 1:3, 1:5, 1:7, 1:9, 1:11 g/mL. After it has reached the desired time, the heating was turned off and cold water was used to cool the reactor until it reaches an ambient temperature. The release valve was opened to lower the reactor pressure to atmospheric pressure as soon as the temperature started to fall. The final product was separated. The resulting solid was dried in the oven for 24 hours at a temperature of 105°C. After that, the hydrochar from the oven was analyzed.

## Chemical Analysis of Sludge and Hydrochar

Analysis was carried out to determine the characteristics of the Hydrochar produced in order to find out the type or class of hydrochar. Moisture content, Ash, Fixed Carbon, and Volatile Matter were determined using Proximate analysis. Ultimate analalysis was determined Carbon, Hydrogen, Nitrogen, Oxygen, and Sulphur content. A bomb calorimeter determined the calorific value. The sludge and hydrochars from HTC process were calculated yield, energy densification and energy recovery by Eq. (1), Eq. (2) and Eq. (3), respectively.

𝑌𝑖𝑒𝑙𝑑 𝐻𝑦𝑑𝑟𝑜𝑐ℎ𝑎𝑟 = 𝑀𝑎𝑠𝑠𝑎 𝐻𝑦𝑑𝑟𝑜𝑐ℎ𝑎𝑟 × 100% (1)

𝑀𝑎𝑠𝑠𝑎 𝑆𝑎𝑚𝑝𝑒𝑙

𝐸𝑛𝑒𝑟𝑔𝑦 𝐷𝑒𝑛𝑠𝑖𝑓𝑖𝑐𝑎𝑡𝑖𝑜𝑛 (𝐸𝑑) = 𝐻𝐻𝑉 𝐻𝑦𝑑𝑟𝑜𝑐ℎ𝑎𝑟

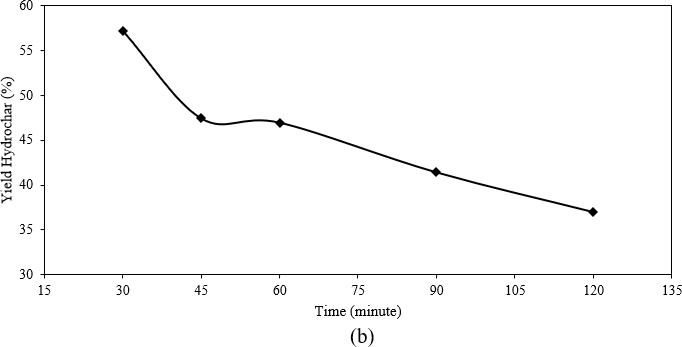
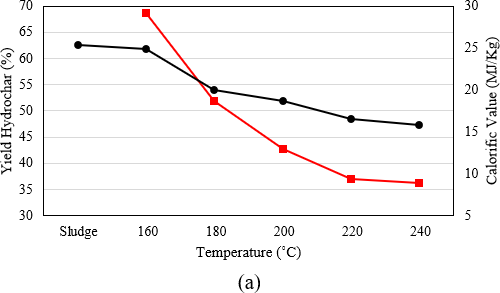
𝐻𝐻𝑉 𝑆𝑎𝑚𝑝𝑒𝑙

(2)

𝐸𝑛𝑒𝑟𝑔𝑦 𝑅𝑒𝑐𝑜𝑣𝑒𝑟𝑦 (𝐸𝑟) = 𝐸𝑛𝑒𝑟𝑔𝑦 𝐷𝑒𝑛𝑠𝑖𝑓𝑖𝑐𝑎𝑡𝑖𝑜𝑛 × %𝑦𝑖𝑒𝑙𝑑 𝐻𝐶 (3)

# RESULT AND DISCUSSION

## The Effect of Temperature and Time Reaction on Hydrochar

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**Figure 1.** (a)The effect of temperature reaction on hydrochar, (b) The effect f time reaction on hydrochar

The effect of temperature reaction of 160 C̊ – 240 C̊ was studied during the hydrothermal carbonization (HTC) of sludge for 120 minutes and 1:9 g/mL of solid-liquid ratio. The reactor processes in subcritical conditions with the addition of water and CO2 gas. Subcritical conditions are conditions where the temperature and pressure are above the boiling point of a solution and below the critical point so that the solution remains in the liquid phase. The

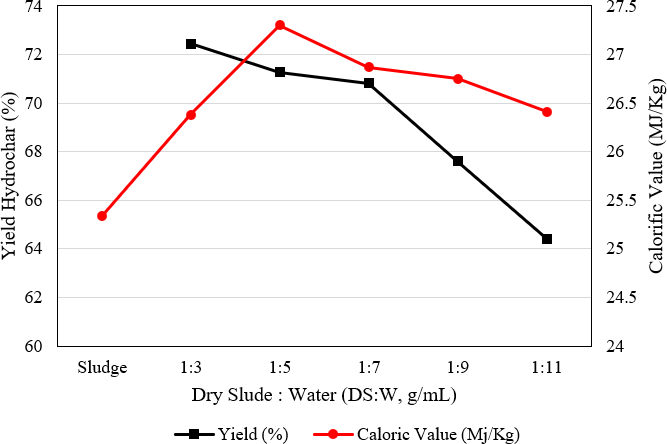
addition of carbon dioxide gas is a substitute for an acid catalyst which during the reactor process can have an acidic effect on the solution. It is evident from **Figure 1(a)**, that the yield of hydrochar will decrease with increasing reactor heat temperature. The decrease of yield is more pronounced at temperatures below 200 C̊ (by 26%) and weakens at temperatures between 200 C̊ to 240 C̊ (by 6.45%). This is consistent Y.Wang, et al (2022) which similarly used subcritical operating conditions and found that raising the reaction temperature gradually reduced the yield of hydrochar [2]. The reaction temperature provides sufficient heat to decompose organic macro molecules for active chemical bond fragmentation and recombination. Therefore, hydrothermal reactions will become more active at higher temperatures and accelerate the rate of degradation and polymerization of sludge compounds [5]. Particularly the increase in temperature reaction promoted the decomposition of macromolecules organic matter, as well as the hydrolysis of inorganic solid matter [2].

The effect of time reaction (30 – 120 min) was studied during the hydrothermal carbonization (HTC) of sludge conducted at 220 C̊ and 1:9 g/mL of solid-liquid ratio. **Figure 1(b)** illustrates, the yield of hydrochar will decrease with increasing reaction time. Upon reaching 45 minutes of reaction time, the yield of hydrochar progressively dropped by 9.77%. On the other hand, the yield of hydrochar only marginally dropped by 0.56% when the reaction time was increased to 60 minutes. The yield of hydrochar dropped 8.81% more when the duration was increased to 120 minutes. Based in the reaction speed equation, reaction time affects the concentration of the mixture. The longer the time, reactant concentration will drop and products will be created [6]. Longer reaction time means higher reaction intensity. Therefore, within a certain reaction time, increasing the reaction time will promote the degradation of organic components and the dissolution of inorganic components in the sludge [2]. The reduced yield is also caused by the availability of sufficient time for decarboxylation and dehydration reactions to transfer the organic matter to the liquid phase. [3].

The calorific value of hydrochar and sludge was evaluated through a bomb calorimeter. It is evident from the calorific value results in **Figure 1(a)** that the calorific value decreased, as the temperature increased. The sludge’s calorific value was 25.34 MJ/Kg; at 160 C̊ , it fell marginally by 0,42 MJ/Kg. The calorific value dropped sharply by

9.1 MJ/Kg when the temperature increased from 160 to 240 C̊ . According to Rather et al (2017), as temperature rises, the yield of hydrochar will decrease while the calorific value will increase due to increased carbonization [3]. The decrease in calorific value in this case may be due to overuse of water in the HTC process, so the influence of solid-liquid ratio must be investigated to raise the calorific value of hydrochar.

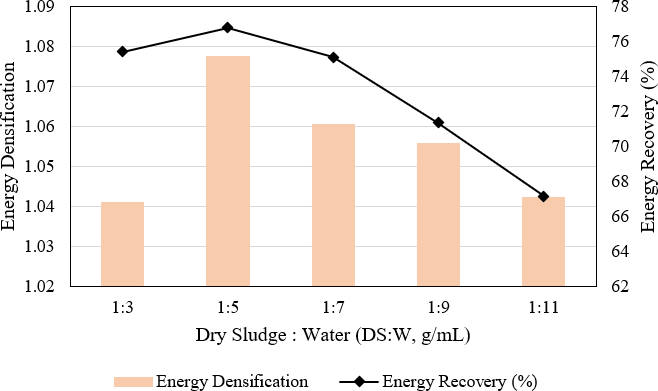
## The Effect of Solid-Liquid Ratio on Hydrochar

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**FIGURE 2.** The effect of solid – liquid ratio on yield and calorific value of hydrochar

The effect of the solid – liquid ratio (DS:W Ratio, 1:3, 1:5, 1:7, 1:9, 1:11 g/mL) in the HTC sludge process was researched at a temperature of 220℃ for 120 minutes. The solid - liquid ratio is a crucial HTC process parameter, since an excessively low water content may result in the reactor overheating and an uneven temperature distribution. However, high water content will also affect the acceleration of the hydrothermal carbonization reaction [7]. **Figure 2** shown, the effect of solid-liquid ratio on yield and calorific value of hydrochar. It is evident from a variety of ratios that the yield of hydrochar will decrease with increasing water addition during the HTC process. The yield of hydrochar decreased by 72,44% (ratio 1:3 g/mL) to 64,41% (ratio 1:11 g/mL). The excess water content in the sludge contributes to yield loss by facilitating the organic matter easily dissolved in the liquid phase and forming a small quantity of residue, the majority of which is mineral dominated [5].

Increased water content in the process will also impact the calorific value, hydrolysis and decarboxylation reactions. As shown in **Figure 2**, the calorific value of sludge, which was initially 25.34 MJ/Kg, increased to 27.3 MJ/Kg (ratio 1:5 g/mL). Nevertheless, there was a drop to 26.41 MJ/Kg (ratio 1:11 g/mL) with the subsequent addition of water. According to He, et al (2015) research, using more water in the HTC process result in a decreased calorific value [7]. The macroalgae *Laminaria Saccharina* experienced a similar drop in calorific value during the HTC process, going from 28.8 MJ/Kg (ratio 1:5 g/mL) to 12.6 MJ/Kg (ratio 1:20 g/mL) [8].



**FIGURE 3.** The effect of solid – liquid ratio on energy densification anergy recovery of hydrochar

The main parameters used to assess the HTC process’s effectiveness are yield of hydrochar, energy recovery and energy densification. Energy densification is a process of increasing the energy density of a material. As the solid – liquid ratio rises, **Figure 3** shown that the energy densification also increases from 1.4 (ratio 1:3 g/mL) to 1.8 (ratio 1:5 g/mL), then decreases slightly to 1.6 at ratios of 1:7 and 1:9 g/mL. At a ratio of 1:11 g/mL the energy densification drops to 1.4. The energy recovery showed a similar pattern, there was an increased from 75.41% (ratio 1:3 g/mL) to 76.79% (ratio 1:5 g/mL) then decreased to 67.14% (ratio 1 :11 g/mL). The lower yield of hydrchar will make low energy recovery. The HTC sludge process which uses an acetic acid catalyst, has a similar behavior where energy recovery increases from 70.41% to 90.31% then decreases to 68.29% [4].

## The Properties of Sludge and Hydrochar

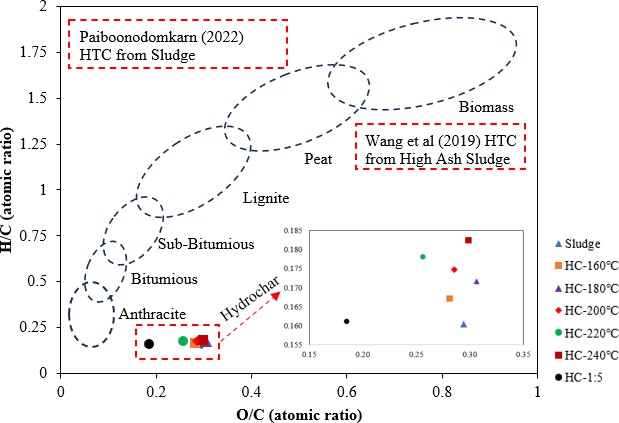
The composition of hydrochar can change during the hydrothermal carbonization process, some of the volatile compounds from organic compounds will dissolve into the liquid phase. The characteristics of the hydrochar and sludge are shown in **Table 2**. Sludge with a high carbon content of 56.46% and low ash content of 16.13% has rather good qualities. The high calorific value of 25.34 MJ/Kg for sludge is in line with the low ash level. The content of C, H, N, and O drops as temperature and time reaction increase, because these components mix into the liquid phase. Carbon steadily drop by 22.37%. Between temperatures of 160 - 180℃, there was a 1.38% decrease in hydrogen, which was subsequently rather stable at 200℃ and slightly decreased by 1% at 240℃. Oxygen steadily drops to 6.43%. The decrease in Oxygen is probably caused by sludge dewatering or protein decarboxylation [9]. The sulfur content of hydrochar remains generally constant at 0.3% - 0.4% as the temperature rises. Meanwhile, there was an increase in ash content along with increasing temperature and time by 32%. The sludge’s proteins release nitrogen, sulfur and oxygen which results in a rise in ash content. This element has an amine group. The compounds left behind in hydrochar are inorganic compounds that are insensitive to temperature and time reaction. Lower levels of nitrogen and sulfur will lessen the likelihood that the chemical compounds NOx and SOx, both of the main air pollutants, will occur. Kim (2014) claims that, because sulfur and nitrogen cannot be eliminated by the hydrothermal carbonization process, the sulfur is highly stable. The hydrothermal temperature must be higher than 220 C̊ to decompose the organic elements N and S [10]. This has been seen in HC-1:5 which was processed by HTC at 220 C̊ , it is evident that nitrogen and sulfur have decreased by 0.1 – 1%. According to Wilk et al (2021), the decomposition of organic materials and mineral retention were also factors in the rise of ash. Excessive water in the process can cause organic compounds to dissolve into the liquid phase, thereby increasing the ash content, reducing the calorific value and carbon content of the hydrochar. This is demonstrated in HC-1:5, where it is evident that the hydrochar’s carbon content is omparatively steady and the ash content is slightly increasing of 6.87%[11].

**TABLE 2.** Properties of sludge and hydrochar

|  |  |  |  |  |  |  |  |  |  |
| --- | --- | --- | --- | --- | --- | --- | --- | --- | --- |
| Variable | Ash% | C% | H% | N% | S% | O% | (O+N)/C | O/C | H/C |
| Sludge | 16.13 | 56.46 | 9.06 | 1.43 | 0.31 | 16.61 | 0.32 | 0.29 | 0.16 |
| HC-160℃ | 24.87 | 50.73 | 8.48 | 1.32 | 0.34 | 14.26 | 0.31 | 0.28 | 0.17 |
| HC-180℃ | 32.43 | 44.72 | 7.68 | 1.12 | 0.34 | 13.71 | 0.33 | 0.31 | 0.17 |
| HC-200℃ | 37.97 | 41.48 | 7.25 | 1.08 | 0.38 | 11.84 | 0.31 | 0.29 | 0.17 |
| HC-220℃ | 44.70 | 37.64 | 6.70 | 0.94 | 0.38 | 9.64 | 0.28 | 0.26 | 0.18 |
| HC-240℃ | 48.13 | 34.09 | 6.22 | 0.98 | 0.40 | 10.18 | 0.33 | 0.30 | 0.18 |
| HC - 1:5 | 23.00 | 56.72 | 9.14 | 0.43 | 0.22 | 10.49 | 0.19 | 0.18 | 0.16 |

There are four different types of coal, namely lignite, sub-bitumious, bitumios, and anthracite as well as two more types of hydrochar made from earlier studies are shown in **Figure 4**. The hydrochar’s polarity, degree of carbonization, and aromaticity are indicated by the (O+N)/C, O/C and H/C. Reduced O/C and H/C ratios suggest a higher aromaticity and degree of carbonization. Meanwhile, a low (O+N)/C ratio denotes a decrease in the hydrochar’s polarity [2]. The Van Krevelen diagram shows the carbonization pathway for the elements C, H and O during the hydrothermal carbonization process. **Table 2**, illustrates how temperature affects polarity and aromaticity by increasing the O/C value and decreasing (O+N)/C. The decrease in C, H and O content suggests that decarboxylation and dehydration reactions are the main reactions in hydrochar formation [12]. Ghanim (2017) states that because the energy of the C element is higher, a smaller O/C number will result in more profitable energy recovery [13]. The degree of carbonization is indicated by the H/C value. In this research, the H/C value slightly increased with increasing temperature and processing time. This indicates that aromatization decreased. The H/C value of hydrochar between 0.16 to 0.18, which is comparible to the Anthrachite coal. Nonetheless, the H/C ratio is comparatively steady at HC – 1:5 [14]. Reporting from the European Biochar Certificate, the hydrochar produced in this research is Bio Carbon Minerals (BCM) because the C value is below 50%. Meanwhile, according to IBI Biochar Standards, the hydrochar in this study is classified as hydrochar type 2 where the C element ranges from

≥30% - <60% [15]. Thus, by optimizing process operating conditions, this hydrochar can be mixed with coal and is considered the main renewable fuel for industrial applications with the best hydrothermal process operating conditions using a temperature of 220 C̊ with a solid:water ratio of 1:5 g/mL for 120 minutes .



**Figure 4.** Van Krevelen Diagram of Hydrochar

# CONCLUSION

This study provides a preliminary exploration of hydrothermal carbonization. The investigation focused on the yield and characteristics of hydrochar, revealing that the yield of hydrochar decreased with increasing reaction temperature and time. This decrease was particularly significant at temperatures below 200°C, with a reduction of 26%. Additionally, the calorific value of the hydrochar decreased as the temperature increased. The sludge’s initial calorific value was 25.34 MJ/kg, but it dropped sharply by 9.1 MJ/kg when the temperature increased from 160°C to 240°C. The high water content in the sludge contributed to the yield loss by making organic matter more likely to dissolve in the liquid phase. The yield of hydrochar also decreased with an increase in the solid-liquid ratio, dropping by 8.03% from a ratio of 1:3 g/mL to 1:11 g/mL. Increased water content during the hydrothermal carbonization process impacted the calorific value, hydrolysis, and decarboxylation reactions. The calorific value increased to 27.3 MJ/kg at a ratio of 1:5 g/mL and then decreased to 26.41 MJ/kg at a ratio of 1:11 g/mL with the subsequent addition of water. Compared to the original sludge, the hydrochar produced exhibited higher aromaticity, a greater degree of carbonization, a higher calorific value, and lower porosity. These characteristics suggest its potential as a solid fuel substitute for coal. The optimal conditions for the hydrothermal process were identified as a temperature of 220°C, a solid-to-water ratio of 1:5 g/mL, and a reaction time of 120 minutes.

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