Rejected red macroalgae (Kappaphycopsis cottonii) as feedstock of bio-oil and biochar: Solid degradation kinetics

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International Journal of Engineering

Rejected red macroalgae (*Kappaphycopsis cottonii*) as feedstock of bio-oil and biochar: Solid degradation kinetics

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ABSTRACT

In the pursuit of renewable energy sources, biomass conversion into biofuels has garnered attention as a sustainable alternative to fossil fuels. This study centers on the pyrolytic conversion of red macroalgae, specifically rejected *Kappaphycus cottonii*, an underutilized biomass in Indonesia, into valuable bio-energy carriers, while analysing the solid degradation kinetics involved. The dried *K. cottonii* underwent slow pyrolysis at varying temperatures (400-600 °C) and time intervals (10-50 min). We assessed the influence of these parameters on bio-oil and biochar yields and detailed mass loss profile using thermogravimetric analysis. The study also explores the chemical kinetics of the decomposition process, utilizing the first-order reaction model to analyze the solid biomass conversion rate, activation energy, and frequency factor. Noteworthy results indicate that an increase in pyrolysis temperature significantly improves bio-oil yield up to a pinnacle at 500 °C, with subsequent reduction due to thermal cracking. Reaction times also greatly affect yield, highlighting the importance of optimized pyrolysis conditions. The activation energy and frequency factor derived from the Arrhenius plot for pyrolysis suggest a lower energy barrier for the decomposition of *K. cottonii* compared to terrestrial biomass, which can be attributed to its distinct chemical composition, especially its low lignin content.

Graphical Abstract
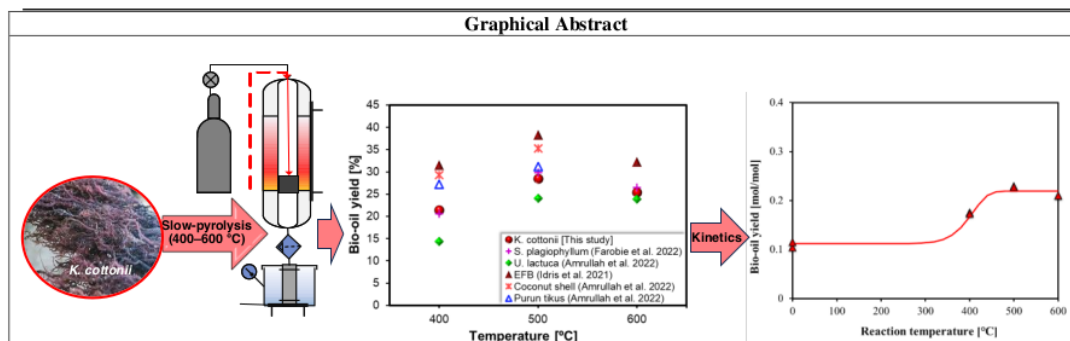
1. INTRODUCTION

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The quest for sustainable and environmentally conscious energy sources has intensified in recent years, spurred by the escalating demand for fossil fuel-derived energy and its associated rise in atmospheric greenhouse gas emissions (1). As the global community grapples

with these challenges, bioenergy emerges as a pivotal component in shifting to a more sustainable framework for energy, potentially mitigating the adverse impacts on our climate and ecosystem (2).

In this context, the exploration of biomass as a renewable and alternative feedstock is gaining attention, posing an intriguing solution to the ever-present energy dilemma. Biomass, derived from plant, harnesses the sun's energy and Earth's nutrients, offering a carbon-neutral alternative to conventional fossil fuels (3). As researchers and industry stakeholders delve deeper into this field, the sustainability of biomass energy production systems comes to the forefront of contemporary conversations. One of the pressing debates centers on the trade-offs between bioenergy production and agricultural demands for food cultivation.

Amid this discourse, aquatic biomass distinguishes itself as a promising candidate, boasting several advantages over land-based biomass sources. Notably, marine and freshwater biomass, such as macroalgae, does not encroach upon valuable arable land needed for food production, sidestepping one of the major hurdles facing terrestrial biomass (4). Apart from that, macroalgae present a myriad of advantages, including enhanced growth velocities and greater yields of organic matter, compared to their terrestrial counterparts. Moreover, these aquatic plants have the capability to flourish in different aquatic environments, from oceans to rivers and even water that has been used, all while eliminating the demand for fertile soil and additional nutrients (5).

The red macroalgae species known as *Kappaphycopsis cottonii*, also referred to as *Eucheuma cottonii*, is extensively farmed in the marine aquaculture of Indonesia. The industrial use of *K. cottonii* is primarily for the extraction of the hydrocolloid carrageenan, which has applications in the food and pharmaceutical industries. Despite its ability to produce large amounts of biomass, a significant quantity is often discarded due to the fact that only 25-35% of it contains carrageenan (6). Consequently, a major portion of this seaweed ends up as solid waste, largely thrown away, contributing to environmental pollution.

Valorizing the discarded biomass for valuable by-products is not only ecologically imperative but also a key step towards advancing a sustainable, circular economy. The overlooked potential of industrial waste from *K. cottonii* can be unlocked by transforming it into bio-oil and biochar through pyrolysis processes. Prior investigations have indicated that applying heat treatment within the range of 400 to 600 °C through pyrolysis can successfully yield bio-oil and biochar

(7,8). Despite its promise, the literature reflects a deficit of research and practical uses for *K. cottonii* residue, particularly post-carrageenan production. Prior investigations have shown effective pyrolysis of various macroalgae types like *Laminaria japonica*, *Undaria pinnatifida*, and *Porphyra tenera*, at 300 to 600 °C, yet these studies did not include *K. cottonii* and focused solely on bio-oil yields without examining the reaction kinetics (9). Our recent work has delved into the decomposition rates during slow pyrolysis of brown and green macroalgae, specifically *Sargassum plagiophyllum* and *Ulva lactuca*, with the aim of extracting bio-oil and biochar (10,11). Saeed et al. conducted experiments using *E. cottonii* through pyrolysis in a tubular furnace under an N_2 atmosphere at 250 to 550 °C for 120 min (12). However, their research was only directed at biochar production with environmental applications in mind, such as absorbing methylene blue dye, and did not consider bio-oil extraction.

The aforementioned research alludes to the potential of *K. cottonii* in producing fuel and chemicals via pyrolysis; however, a comprehensive analysis of the pyrolysis kinetics of *K. cottonii* remains absent from the current body of scholarly work. This gap highlights the originality and critical nature of our research endeavor. A thorough kinetic analysis is imperative to glean critical insights into the degradation behavior of this macroalgae, including its rate of decomposition and the factors influencing it. Such information is vital, laying the groundwork for the design and optimization of pyrolysis reactors, thereby enabling a more efficient and commercially viable transition from laboratory to industry-scale operations.

In this study, we investigate the degradation kinetics of *Kappaphycopsis cottonii* under pyrolytic conditions, meticulously calculating its rate of decomposition, activation energy, and pre-exponential factor. These parameters will not only aid in the prediction of conversion processes and elucidation of reaction mechanisms but will also inform future endeavors to rehabilitate this underutilized biomass into a cornerstone of renewable energy production.

2. MATERIALS AND METHODS

2.1. Biomass provision
The *K. cottonii* macroalgae waste used in this work were obtained as a byproduct from PT. Bantimurung, a carrageenan producer located in South Sulawesi. These macroalgal biomass were subjected to a drying process at 105 °C for 12 h to reduce the water content. Subsequently, the dried material was processed through grinding and

passed through a sieve to attain a size of 60 mest for uniformity in the samples.

2.2. Pyrolysis procedure   The feedstock of *K. cottonii* was subjected to thermal degradation within a single-batch reactor system, previously detailed in our related publications (10,13,14). In summary, the reactor, constructed from stainless steel and incorporating an electrically powered furnace, was fitted with a thermocouple for temperature measurement, a PID temperature controller for precise thermal regulation, and a condenser. Before initiating the pyrolysis, an inert atmosphere was established within the reactor by flushing it with nitrogen gas to expel ambient air and oxygen. The experimental pyrolysis was conducted across a spectrum of temperatures from 400 to 600 °C and were timed at different intervals spanning from 10 to 50 min. Post each experimental run, the reactor was allowed to cool. Following the pyrolysis, bio-oil was collected and separated from the solid residuals. To ensure accuracy, each preparatory phase was replicated to verify the consistency of bio-oil measurements. The bio-oil yield was determined through the proportion of the mass of bio-oil produced relative to the original mass of the *K. cottonii* used. Meanwhile, the production of biochar was quantified by noting the change in mass of the quartz reactor pre and post-pyrolysis. Additionally, gaseous byproducts were quantitatively assessed based on the weight discrepancy between the yields of bio-oil and biochar.

2.3. Product analysis   The stability to heat of both *K. cottonii* raw material and the resultant biochar was analyzed through thermogravimetric analysis using a TGA 4000 instrument from Perkin Elmer. An 8 mg sample of the *K. cottonii* feedstock was placed into an alumina pan for assessment. The evaluation tracked the weight changes of the samples with heat application from ambient temperature up to 800 °C at a rate of 10 °C per minute. To maintain an inert atmosphere throughout the test, nitrogen gas was continuously flowed through at a rate of 50 mL per minute.

3. RESULTS AND DISCUSSION

3.1. Influence of temperature on Bio-oil Yield
The relationship between pyrolysis temperature and bio-oil yield derived from *K. cottonii* was studied in detail. **Figure 1** illustrates the variation in bio-oil output correlated with different temperature settings and processing times during the controlled thermal decomposition of *K. cottonii*. It was observed that higher temperatures played a pivotal role in increasing bio-oil yields. A marked improvement in yields was noted when the temperature conditions were elevated from 400 to 500 °C, likely due to the intensified

breakdown of the macroalgal biomass. Furthermore, a rise in temperature may also contribute to the more extensive degradation of the resulting solid byproduct, biochar. Despite this, an uptrend in bio-oil production did not continue when the temperature reached 600 °C, which may be ascribed to the bio-oil undergoing thermal decomposition into gaseous substances at temperatures above 500 °C. These observations align with the results documented by Hao et al. (15) and Aboulkas et al. (16), confirming the trend where bio-oil yields climb with an increase in temperature up to 500 °C before declining.

The duration of the reaction also significantly impacted the yields of bio-oil from pyrolyzing the red algae *K. cottonii*. Initially, yields were minimal, with only 12.05% yield recorded at 400 °C for a 10-minute reaction. This low yield could be related to the incomplete pyrolytic process at abbreviated reaction times and reduced temperatures (17). However, extending the pyrolysis period from 10 to 50 min led to a substantial yield increase from 12.05 to 21.38% at 400 °C. A similar pattern was observed at 500 °C, where there was an increase in the bio-oil yield from 16.32 to 28.51% in correlation with the extension of the reaction time.
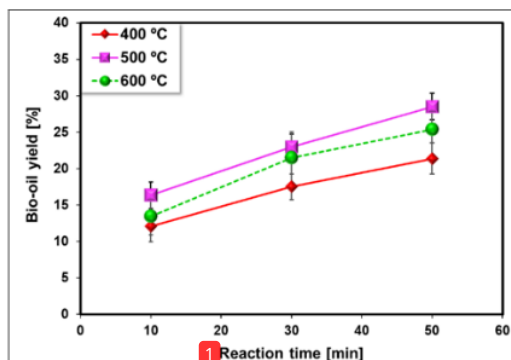

Figure 1. The impact of temperature and duration on the bio-oil yield during the gradual pyrolysis process of *K. cottonii*

Comparative studies of bio-oil yields were also conducted with other biomass sources, such as *Ulva lactuca* (11), *Sargassum plagiophyllum* (10), and terrestrial feedstocks like coconut shell (19), purun tikus (20), and empty fruit bunch (EFB) (18). **Figure 2** presents a comparative yield analysis between *K. cottonii* and various other biomass types. The findings revealed that the production of bio-oil from *K. cottonii* was generally lower than those extracted from terrestrial sources like EFB, coconut shells, and purun tikus. This variance in yield might be attributed to differences in lignin content, with macroalgae typically having less

lignin than terrestrial plants (21,22). Biomass that contains a greater proportion of lignin has been linked to greater bio-oil yields, along with a higher concentration of phenolic compounds, as noted by Klemetsrud et al (23). Interestingly, the bio-oil yield from *K. cottonii* surpassed that of *U. lactuca*, which could be due to the former's higher carbohydrate constitution. *K. cottonii* has been characterized by a carbohydrate content of $61.90 \pm 0.12\%$, proteins making up $4.77 \pm 0.08\%$, and lipids at $1.72 \pm 0.04\%$, in contrast to *U. lactuca*'s respective contents of $40.66 \pm 0.14\%$, proteins at $22.92 \pm 0.21\%$, and lipids at $1.32 \pm 0.06\%$ as detailed by Hartulistiyoso et al. (24).
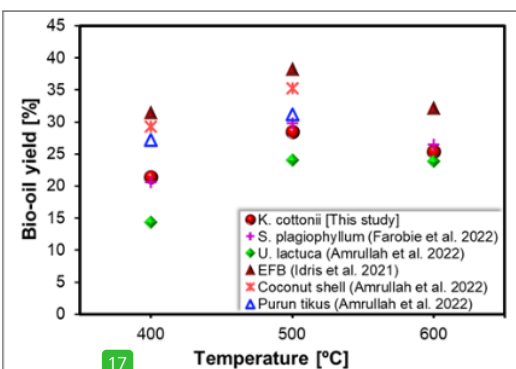

Figure 2. The bio-oil yield obtained from the pyrolysis of *K. cottonii* in comparison with other biomass sources.

3.2. The impact of varying temperatures on solid decomposition
To investigate the appropriate temperatures for the pyrolysis of *K. cottonii*, both the raw biomass and its derived biochar were subjected to a thermogravimetric analysis (TGA) to monitor the mass loss as temperature increases. **Figure 3** displays the TGA curves for both the *K. cottonii* biomass and its biochar. Notably, the *K. cottonii* exhibited significant weight reductions at temperatures of 259 ºC and 421 ºC, with mass decrements of approximately 13.5% and 54.5%, respectively. These substantial losses are primarily attributed to the de-volatilization of organic matter. As reported by Kim et al., weight loss in biomass typically signifies the decomposition of macro molecules, such as proteins and carbohydrates (25). Beyond 600 ºC, the *K. cottonii* experienced a dramatic loss of around 64.7%, which is likely due to the disintegration of its inorganic components, notably metal-based compounds.

It is noteworthy that more than half of the *K. cottonii* mass dissipated at temperatures exceeding 387 ºC, indicating the necessity of conducting pyrolysis at temperatures above this threshold. Turning to the biochar, negligible mass change was noted up to 350 ºC. Nonetheless, there was roughly a 12.4% reduction in the mass of biochar at 400 ºC, which can be explained by the resilience of its primary component, polyaromatic structures, which are more resistant to breakdown compared to proteins and carbohydrates (26). When exposed to 600 ºC, biochar underwent a reduction in mass of approximately 24.1%, illustrating that higher temperatures lead to thermal decomposition into gaseous products.
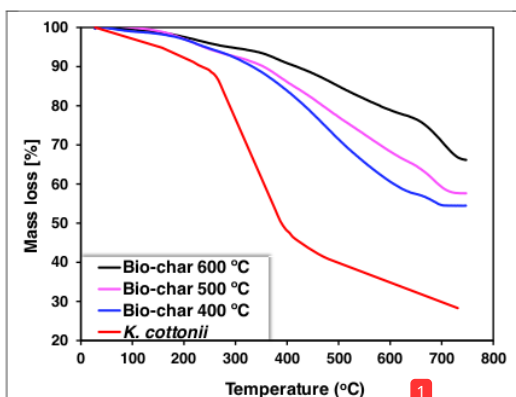

Figure 3. Thermal properties of *K. cottonii* and its corresponding bio-char using thermogravimetric analysis

Furthermore, the morphological changes in red macroalgae *K. cottonii* induced by temperature are consisten with the TGA study. **Figure 4** features the visual transformations of the fresh algae in contrast to the resultant biochar. The original macroalgae condition is pictured in Fig. 4(a), displaying apparent debris. Subsequently, Fig. 4(b) depicts the *K. cottonii* post washing, and Fig. 4(c) presents the algae post-drying and grinding, achieving a consistent particle size near 0.25 mm. Upon pyrolysis at temperatures between 400 and 500 ºC, a darkening to black of the algae's coloration is observed, whereas at 600 ºC, the color shifts to a more brownish-black hue, signifying a process that yields a high carbon content material. Through the slow pyrolysis approach undertaken, biochar is generated as the primary product, with yields ranging between 40.72-76.45%.

3.3. Reaction kinetics of solid degradation
Parameters indicating the rate of reaction provide crucial understanding into the transformation dynamics of solid biomass throughout the pyrolysis procedure. In the scenario of *K. cottonii* pyrolysis, a model representing the solid state decomposition during the gradual pyrolysis is showcased in **Figure 5**.
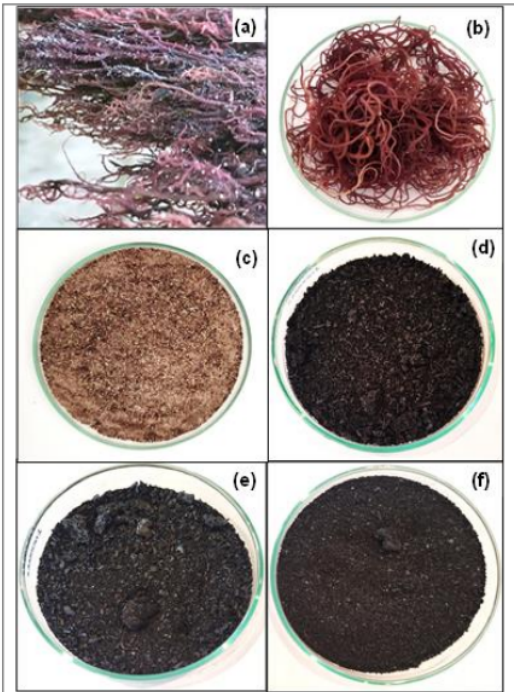
Figure 4. The physical appearance of (a) fresh specimens collected from the beach (b) algae post-wash with water, (c) algae after being oven-dried and ground, (d) biochar at 400 °C, (e) biochar at 500 °C, and (f) biochar at 600 °C
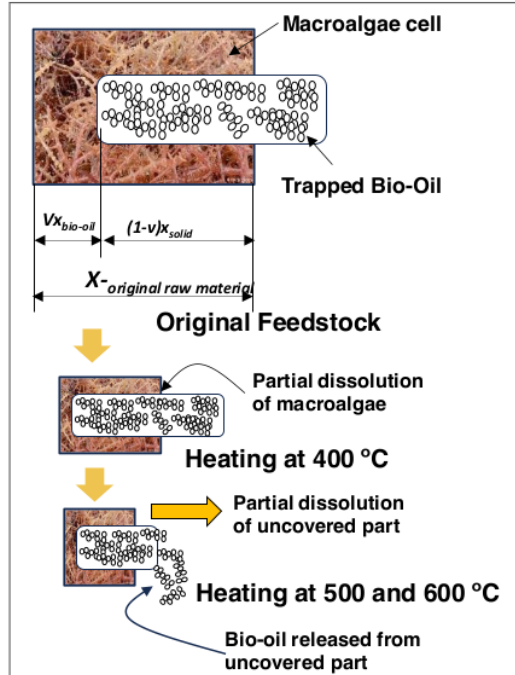
$$\frac{d[X_{cottonii}]}{d_t} = -k[X_{cottonii}] \tag{2}$$

Figure 5. Formulation depicting the degradation mechanism of solids throughout the gradual pyrolysis of *K. cottonii*

Given that the solid degradation process aligns with a first-order kinetic reaction, it is possible to ascertain both the decomposed solid's proportion and the kinetic constant for the reaction. **Figure 5** displays a graphical representation of the solid degradation pathway, from which a corresponding mathematical formula can be derived as:

$$X_{cottonii} \rightarrow vX_{bio-oil} + (1-v)X_{solid} \tag{1}$$

In this context, $X_{cottonii}$ denotes the initial proportion of the *K. cottonii* feedstock. The variable v is used to express the portion of the solid that converts into bio-oil. $X_{bio-oil}$ and X_{solid} are defined as the fractions of bio-oil produced and the remaining solid material, correspondingly. With the presumption of a first-order kinetic process, it is possible to establish the differential rate expression for the conversion of *K. cottonii* at any given moment, t, through the following formula:

Taking into account the principles of mass conservation, the balance equation can be articulated in this manner:

$$\begin{aligned}[X_{cottonii}] + [X_{liquid}] + [X_{solid}] \\ = \text{constant} = [X_{cottonii}]_i + [X_{liquid}]_i \\ + [X_{solid}]_i = [X_{cottonii}]_t \end{aligned} \tag{3}$$

In this notation, '*i*' signifies the beginning value, and '*t*' stands for the cumulative quantity of *K. cottonii* encountered throughout the pyrolysis process. In a similar vein, the quantity of $X_{cottonii}$ is quantifiable and can be outlined as follows:

$$[X_{cotoonii}] = \frac{1-v}{v}[X_{liquid}] \tag{4}$$

By integrating Equation (4) into Equation (3), we can articulate the mass balance equation in the following way:

$$[X_{cottonii}] = [X_{cottonii}]_t - [X_{liquid}] -$$
$$[X_{solid}] = [X_{cottonii}]_t - [X_{liquid}] -$$
$$\frac{1-v}{v}[X_{liquid}] = [X_{cottonii}]_t -$$
$$\left(1 + \frac{1-v}{v}\right)[X_{liquid}] = [X_{cottonii}]_t -$$
$$\frac{1}{v}[X_{liquid}] \qquad (5)$$

Subsequently, by replacing Eq. (5) into Eq. (2), it becomes possible to ascertain the rate of change, which is delineated as follows:

$$\frac{d}{dt}\left([X_{cottonii}]_t - \frac{1}{v}[X_{liquid}]\right) =$$
$$-k\left([X_{cottonii}]_t - \frac{1}{v}[X_{liquid}]\right) \qquad (6)$$

Equation (6) may be reformulated as:

$$\frac{d}{dt}([X_{liquid}]) = vk\left([X_{cottonii}]_t - \frac{1}{v}[X_{liquid}]\right) \qquad (7)$$

Conversely, the yield of the liquid product can be articulated in the following manner:

$$Y_{liquid} = \frac{[X_{liquid}]}{[X_{cottonii}]_t} \qquad (8)$$

Therefore, Eq. (7) can be formulated as:

$$\frac{d}{dt}Y_{liquid} = vk - kY_{liquid} \qquad (9)$$

Eq. (9) can be recast in the following form:

$$\frac{dY_{liquid}}{vt - kY_{liquid}} = dt \qquad (10)$$

$$ln(v - Y_{liquid}) = -kt + C \qquad (11)$$

$$Y_{liquid} = v - A\,exp(-kt)) \qquad (12)$$

The rate constant is derivable using the Arrhenius formula, detailed as:

$$k = A\,exp\left(\frac{E_a}{RT}\right) \qquad (13)$$

where, $'T'$ dnotes the temperature, $'E_a'$ is the activation energy, $'A'$ denotes the frequency factor, and $'R'$ is the universal gas constant. Subsequent to incorporating the Arrhenius expression into Equation (12), the revised formula is presented as:

$$Y_{liquid} = v - \left(v - Y_{liquid,i}\right)exp\left(-A\,exp\left(\frac{E_a}{RT}\right)t\right) \qquad (14)$$

The least-squares error (LSE) approach was employed to calculate both the reaction rate constant and the percentage of solid material that undergoes transformation into the liquid phase. The graphical representation in **Figure 6** demonstrates the correlation of the experimental data with the theoretical value depicting the variation of temperature in relation to the yield of bio-oil. The close alignment of the experimental results with the theoretical curve was evident.
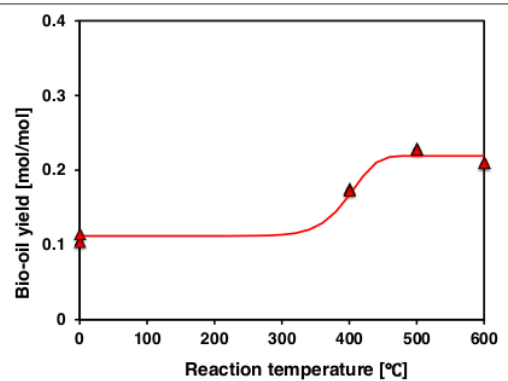

Figure 6. The curve-fitting analysis illustrating the yield of bio-oil at various temperatures encountered during *K. cottonii* pyrolysis

This was further substantiated by the parity diagrams which exhibited a high coefficient of determination (R^2), specifically 0.9958, as illustrated in **Figure 7**. **Figure 8** depicts the Arrhenius graph that details the decomposition rate of *K. cottonii* during the pyrolysis process. Both the activation energy and the frequency factor were extrapolated from this graph. The frequency factor specific to the pyrolysis of *K. cottonii* is estimated at approximately 0.362 s⁻¹. Moreover, the activation energy calculated for the pyrolysis of *K. cottonii* is around 24.30 kJ mol⁻¹, which is significantly less when compared to the values reported in studies involving land-derived biomass. Genieva et al. found a reported activation energy of approximately 125.3 kJ mol⁻¹ for almond shell pyrolysis (27). Similarly, Amrullah et al. identified the activation energy for the pyrolysis of

coconut shell to be in the region of 153 kJ mol^{-1} (19). The discrepancy in these values can be attributed to the varied chemical compositions found within different biomass types. Notably, macroalgae exhibit a reduced lignin content and are predominantly composed of carbohydrates compared to arable-land plants.\, resulting in a reduced energy requirement for macroalgal decomposition compared to that of terrestrial biomass.
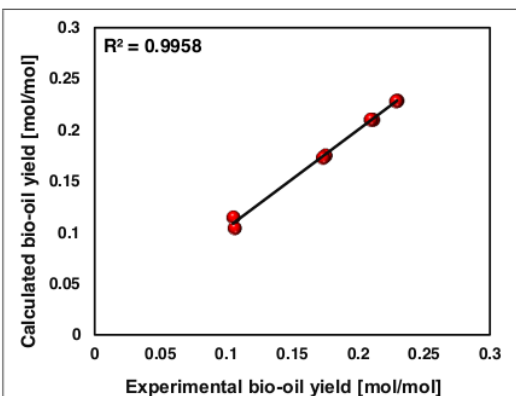

Figure 7. Plots of parity showcasing a comparison between bio-oil yields derived from experimental measurements and those predicted through calculations
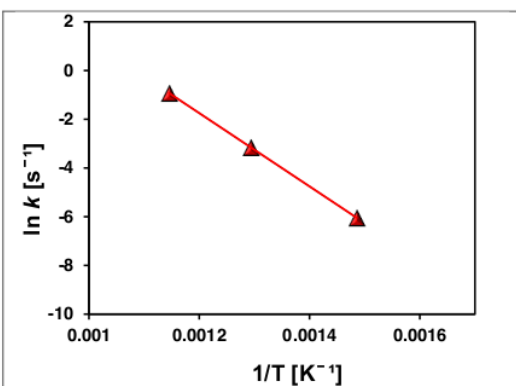

Figure 8. Graphical representations according to Arrhenius for the thermal decomposition of *K. cottonii*

4. CONCLUSION

This work has successfully showcased the potential of utilizing rejected red macroalgae, specifically *Kappaphycus cottonii* residues, as a sustainable feedstock for the generation of biofuel through the process of slow pyrolysis. Our findings reveal that temperature and time are critical parameters influencing bio-oil yields, with optimal production observed around 500 °C. We have substantiated that the first-order kinetic model aptly describes the solid degradation kinetics of *K. cottonii* during pyrolysis, resulting the pre-exponential factor of 0.477 s^{-1} and the activation energy of 24.30 kJ mol^{-1}. The activation energy of *K. cottonii* thermal degradation is significantly lower than those for terrestrial biomasses like coconut and almond shells, reflecting the lower energy required for macroalgal degradation due to its chemical composition—predominantly carbohydrates with minimal lignin content. Future research directions may involve exploring the functional applications of produced biochar and extending the kinetic study to other macroalgae species, potentially broadening the scope of macroalgal biomass as a key player in the bioenergy sector.

5. ACKNOWLEDGEMENT

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Persian Abstract

چکیده

در تعقیب منابع انرژی تجدیدپذیر، تبدیل بیوماس به بیو-روغن و بیو-زغال اختلاف جذب برای جایگزین پایدار به سوخت‌های فسیلی به دست آورده است . که یک بیوماس کم استفاده شده در اندونزی است، به حاملان Kappaphycus cottonii این مطالعه بر روی تبدیل پیرولیتیک جلبک‌های قرمز، به ویژه خشک و زمینی شده، K. cottonii. انرژی بیولوژیک ارزشمند تمرکز دارد، در حالی که سینتیک تجزیه جامدات مرتبط را تجزیه و تحلیل می‌کند پیرولیز کند در دماهای متغیر)۴۰۰-۶۰۰ درجه سانتی‌گراد (و فواصل زمانی)۱۰-۵۰ دقیقه (را تجربه کرد .تأثیر این پارامترها بر روی عملکرد بیو-روغن و بیو-زغال و نمودار از دست رفتن جرم را با استفاده از تجزیه و تحلیل گرماگرافیمتریک ارزیابی کردیم .این مطالعه همچنین به تجزیه شیمیایی فرآیند تجزیه پرداخت و با استفاده از مدل واکنش درجه اول برای تجزیه نرخ تبدیل بیوماس جامد، انرژی فعالسازی و عامل فرکانس تحلیل شد . نتایج قابل توجه نشان می‌دهد که افزایش دمای پیرولیز عمده بیو-روغن را تا یک نقطه اوج در ۵۰۰ درجه سانتی‌گراد بهبود می‌بخشد، با کاهش بعدی به دلیل شکست حرارتی .زمان‌های واکنش نیز به شدت تأثیر می‌گذارد، که اهمیت شرایط بهینه‌سازی پیرولیز را برجسته می‌کند .انرژی فعالسازی و عامل نسبت به بیوماس‌های زمینی وجود K. cottonii فرکانس مشتق شده از نمودار آرنیوس برای پیرولیز نشان می‌دهد که مانع انرژی کمتری برای تجزیه دارد، که می‌توان آن را به ترکیب شیمیایی متمایز آن، به خصوص محتوای کم لیگنین آن نسبت داد.

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