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Biochar and Biofuel from Pyrolysis of Oily Rags

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Abstract

In the paper, we present for the first time an experimental study on the pyrolysis of oily rags collected from an automobile factory. The performance of pyrolysis to recycle fresh and used wipes to valuable liquid and solid products was compared. The presence of waste lubricant oil (WLO) on fabric drastically changed the outcome of the process. The specific surface area of pyrolytic char was initially reduced, but can expand to 1178 m^2/g after activation. A very high fraction of oily rags (75.03 wt.%) was converted to high heating value pyrolytic oil (30.3 MJ/kg), resulting in 22.73 MJ recovered from one kilogram of raw material-higher than from most other waste reported. Gas emission from pyrolysis of WLO contaminated wipes was only 6.09 wt.% of raw material- nearly twentyfold less than incineration and ninefold less than pyrolysis of uncontaminated wipes. Overall, pyrolysis of oily rags produced a considerable amount of valuable material and fuel while releasing little greenhouse gases to the atmosphere, making it an eco-friendly and economically feasible solution for this kind of industrial waste.

Keywords: Activated carbon, oily rags, pyrolysis, pyrolytic oil, wipes.

1. Introduction

Petroleum has long been the most important non-renewable resource for fuel and chemical production. The poor estimation and uneven distribution of oil reserves caused the fear of oil shortage in many countries, which in turn spurred the research and policies on energy saving and alternative resources. An important product from petroleum is lubricant oil - or simply oil in daily speaking-with millions of tons produced annually and a market valued at USD164.9 billion in 2021 [1]. Since bio-lubricant account for less than 2% of the global market [1], fossil-derived lubricant, which requires non-renewable resources and takes nearly ten-fold longer than bio-lubricant for degradation [2], still dominates in the coming years. As alternatives are inadequate, petroleum consumption for lubricant production remains high and recycling becomes the salvation, which saves not only the resource but also the environment.

The United States Department of Energy reported that more than 14 million cubic metres of waste lubricant oil (WLO) were commercially collected in 2017, of which 27% was regenerated or close recycled [3]. Italy regenerated circa 190 kilotons of WLO in 2021, while more than 241 million tons of WLO were recycled in South Korea in 2020 [1]. When regeneration and close recycling are not possible, WLO can be converted to fuels, syngas, char, etc. by various technologies such as distillation, solvent extraction, gasification, pyrolysis, hydrolysis, etc., or burned for heat in a cement kiln or waste incinerator [1]. These technologies, except incineration, target liquid WLO collected during repair, maintenance, etc. However, when lubricant oil leaks, it is usually collected using solid material, such as wiping cloth for liquid leakage or filter paper for gas leakage. This results in solid wastes containing WLO with carcinogenic compound such as polychlorinated biphenyl or polycyclic aromatic hydrocarbons. The solid form and low WLO content make these types of waste unsuitable for most of the aforementioned technologies, leaving incineration the only popular choice. Incineration is nevertheless not a treatment method but a disposal method of energy recovery: instead of burying tons of waste, we vent tons of greenhouse gases and possibly carcinogenic byproducts if proper treatment is not implemented.

Concerning the universal consumption of wiping cloths in factories, a treatment that is more eco-friendly than incineration should be applied for it. While textile reuse and recycling have been widely adopted throughout the world, many techniques employed, such as washing, dissolution, digestion, fermentation, composting, etc., require rather "clean" waste than the heavily contaminated ones like rags. This is one of the

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reasons that, despite a diverse range of recycling methods, 75% of global textile waste is landfilled [4]. For fabric contaminated with hazardous compounds like oily rags, mechanical recycling to insulation or construction materials is unsafe, so tertiary recycling approach involving pyrolysis and hydrolysis is the most appropriate. During and after the COVID-19 pandemic, the amount of bio-hazard textile waste increased sharply, with waste masks amounting to more than 2.6 kt/y [5]. Studies on fabric pyrolysis also flourished, with target products being activated carbon [6], fuel [7], or other valuable chemicals [8].

Although many studies have tried to recycle the huge amount of WLO and waste fabric, none of them focus on oil-contaminated fabric like oily rags. Spending on rags is much less than other production cost, created an illusion that their environmental impact is negligible but a simple calculation will prove the opposite: statistic from an automobile factory in Vietnam show that assembling an automobile uses nearly 200 grams of rags, deductively assembling more than 90 million ones as the whole world did in 2023 used about 18 kilotons of rags - seven times the amount of waste mask mentioned above. Taking into account that oily rags from factories have been collected and strictly managed according to national regulations in most countries, an efficient recycling technique can be easier to incorporate into current treatment than other municipal or agricultural wastes.

In this paper, we present for the first time an experimental study on the pyrolysis of oily rags collected from factory. Although experiments on oil and rag pyrolysis have been independently published [1, 9], the co-pyrolysis outcome will be non-trivial because components of oil, rag, and their pyrolysis products can react with each other, especially between the hydrocarbon and the highly oxygenated compounds from natural fibre. Furthermore, the metal added to the lubricant oil during production may have catalytic effects on the pyrolysis of the fabric and alter the properties of pyrolysis products. To investigate the effect of WLO on fabric pyrolysis, fresh wipes are also pyrolyzed to compare with the contaminated ones. The experimental result showed a significant difference between the two materials.

2. Materials and Methods

2.1. Materials

Fresh and used oily rags were collected from an automobile factory in Vietnam. The factory assembled different types of vehicles (SUV, van, pickup truck) from finished parts: engine, transmission, tires, etc. Operations included joining processes (welding, brazing, soldering, and mechanical fixing), painting, and finishing steps. Other metalworking, such as cutting and forming, is not carried out significantly. Wipes were used primarily for cleaning machines, tools, materials, and products during production and maintenance. The collected samples do not include other oil-contaminated fabrics, such as protective cloth (like gloves).

The wipes were made of scrap from apparel manufacturing, an action of reusing post-industrial textile waste. The material is a blend of cotton and synthetic fibre - cotton absorbs lubricant oil and synthetic fibre adds mechanical strength to the wipes. Since the rags were used to wipes lubricant oil off surfaces, not soaked into it, the amount of oil on the rags was about 10% of the total mass - quite small compared to the adsorption capacity of textile specifically designed for oil adsorption as reported in literature [10]. The collected samples (fresh and used ones) are stored in a dark, closed container, at room temperature. The storage conformed to Vietnamese regulations on industrial waste and prevented significant change of the samples, such as evaporation or oxidation.

2.2. Pyrolysis

The experimental setup for pyrolysis is illustrated in Fig. 1.

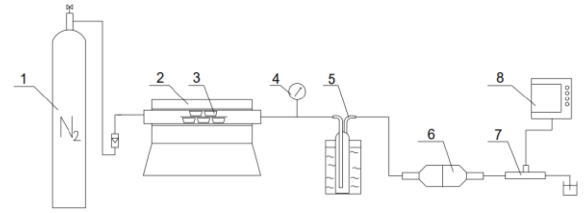

Fig. 1. Pyrolysis experiment setup

(1. Nitrogen cylinder, 2. Furnace, 3. Reactor, 4. Pressure gauge, 5. Condenser, 6. Damper, 7. Sampler, 8. Gas analyser)

The samples were cut into small pieces, 2–3 mm wide and 10–15 mm long and put in combustion boats. Approximately 15 grams of rag were pyrolyzed in a tubular reactor made of stainless steel. The reactor, whose inner diameter is 55 mm and length is 1000 mm, was put horizontally in a Lenton tube furnace model LTF 12/75/750 and fed by a nitrogen flow at a flow rate of 110 mL/min. Once the air was swept out, the temperature was raised from ambient to 500 °C in 25 minutes and kept constant for one hour. This temperature was reported in a previous study [6] where polyester and cotton - the two common components of textiles-were successfully pyrolyzed. In another study [11], a wide range of pyrolysis temperatures from 500 °C to 900 °C was investigated. The higher the temperature, the lower total solid and liquid yield. Since biochar and biofuel are targeted products, 500 °C is the suitable pyrolysis temperature. The duration of one hour is the minimum reported in previous studies [6, 12, 13]. Pressure was kept at 1 atm to minimise both inward and outward leakage. Gas effluent was condensed at 0 °C, then absorbed by water before being vented. Between the condenser and absorber, gas effluent composition was measured by a Gasboard-5020 automobile emission gas analyser, which operated at ambient temperature. The device is based on dual beam non-dispersion infrared principle to measure carbon monoxide, carbon dioxide, hydrocarbons, and electrochemical sensors to measure oxygen and nitrogen oxide. The resolution of oxygen, carbon monoxide, and carbon dioxide measurement is 0.01% and the resolution of nitrogen oxide and hydrocarbons measurement is 1 ppm.

The mass of raw material – M, solid product – S, liquid product – L were measured by analytical balance. Product yields were calculated by the equations:

$$\text{Solid yield: } Y_S = \frac{S}{M}.100\% \tag{1}$$

$$\text{Liquid yield: } Y_L = \frac{L}{M}.100\% \tag{2}$$

$$\text{Gas yield: } Y_G = \frac{M-S-L}{M}.100\% \tag{3}$$

To ensure accuracy, the pyrolysis experiment was repeated numerous times until the average yields could be estimated with standard errors around or less than 0.5%.

2.3. Activation

The solid product of pyrolysis (char) was processed to activated carbon by the following procedure:

- The char was washed by acetone once, then by distilled water three times.
- The washed char was dried at 105 °C in 12 hours, then mixed with potassium hydroxide at a char/KOH ratio of 1/3.
- The mixture was put into distilled water and stirred to form a suspension at a concentration of 0.125 g/mL.
- The suspension was dried at 120 °C for 24 hours and put back in the reactor and fed by a nitrogen flow at a flow rate of 110 mL/min.
- After air was swept out, the temperature was raised from ambient to 550 °C in 25 minutes and kept constant for 90 minutes.
- The product was mixed with excessive hydrochloric acid, then thoroughly washed with distilled water.
- The activated carbon was dried at 105 °C before specific surface area measurement (BET measurement).

2.4. Ex-situ Analytical Methods

The heating value of the liquid products was measured on Parr 1266 Bomb Calorimeter. A controlled amount of liquid product was burned completely together with a controlled amount of copper wire and cotton thread inside the bomb. By measuring the temperature of the controlled amount of water surrounding the bomb, the heat released by the reaction was measured. The heating value of liquid products was measured after subtracting the heat released by burning copper wire and cotton thread.

Specific surface area of the chars and activated carbon was measured on Micromeritics Gemini VII 2390. The measurement took place at a temperature of 77 K, pressure of 770 mmHg, and in the nitrogen flow at a flow rate of 25 mL/min. All samples were vacuum dried at 300 °C for 1 hour before the BET measurement.

3. Results and Discussion

3.1. Product Yields

The pictures of char, activated carbon, and pyrolytic oil are shown in Fig. 2. Tables 1 and 2 show yields from fresh and used oily rags pyrolysis, respectively. For each material, more than 100 grams were pyrolyzed in 8 batches. After activation, the weight of char was reduced by 10%. Taking into account material loss due to multiple times of washing and transferring between containers, the activation yield is nearly 100% and the mass lost due to activation is negligible.


(a) (b) (c)

Fig. 2. Products from oily rags: (a) char; (b) activated carbon; (c) pyrolytic oil

Compared to fresh ones, the solid yield from oily rags was relatively about 10% less, corresponding to the decrease of fabric fraction in the sample, which was estimated from weight of consumed wipes and produced oily waste measured in the factory. In comparison with pyrolysis of cotton fabric at the same temperature [14], the solid yields from both materials are higher. This is owing to the higher carbon content of polyethylene terephthalate (PET) and WLO. Previous studies on uncatalyzed textile pyrolysis at lower and higher temperatures reported higher and lower solid yield, respectively. This matched the common trend of pyrolysis: the higher the temperature is the less solid residue. Hence, there is no sign that the presence of WLO has any exceptional effect on the solid yield.

Table 1. Yields from fresh rags pyrolysis, %

No.	Solid yield	Liquid yield	Gas yield
1	22.08	24.76	53.16
2	20.64	24.28	55.08
3	22.25	23.23	54.52
4	21.05	25.09	53.86
5	20.04	23.57	56.39
6	20.25	23.19	56.56
7	22.42	24.62	52.96
8	22.27	24.87	52.86
Avg.	**21.38 ± 0.35**	**24.20 ± 0.27**	**54.42 ± 0.52**

Table 2. Yields from oily rags pyrolysis, %

No.	Solid yield	Liquid yield	Gas yield
1	18.40	76.00	5.60
2	18.77	74.71	6.52
3	18.69	74.85	6.46
4	19.55	74.26	6.19
5	19.00	74.61	6.39
6	19.50	73.80	6.70
7	18.01	77.00	4.99
8	19.00	74.98	6.02
Avg.	**18.88 ± 0.18**	**75.03 ± 0.36**	**6.09 ± 0.20**

On the other hand, the yields of liquid and gas products varied greatly from fresh to used oily rags: liquid yield increased threefold from 24.20 wt.% to 75.03 wt.% while gas yield decreased ninefold from 54.42 wt.% to 6.09 wt.%. Although the liquid product may contain some components of WLO evaporated prior to real pyrolysis, this phenomenon alone cannot generate so much additional liquid yield because of the low fraction of WLO in the oily rags. It then can be concluded that the presence of WLO has hampered the secondary decomposition of volatile components to non-condensable gas in the fabric pyrolysis by several means: WLO increased heat conductivity of the material, preventing excessive superheating that promote secondary decomposition; hydrocarbons in WLO reacted with oxygen content of the fabric, preventing the formation of carbon oxides; the metal additive in WLO may have some catalytic effect on the pyrolysis, favouring primary decomposition rate over secondary decomposition rate. The shift from gas to liquid product is both scientifically and economically significant. Gas product is usually less desirable from waste pyrolysis due to a larger volume and less valuable components, such as carbon oxides. In a recent review [15], only a study on the pyrolysis of acrylic textile report gas yield less than 10 wt.% after utilising two consecutive dry ice cooled condensers at such a low temperature that propane can condense. Other reported gas yields were mainly in the range of 30 – 50 wt.%, with some exceptions at ~80 wt.% in gasification. In this study, the presence of WLO helped reduce gas yield from 54.42 wt.% to 6.09 wt.%. This reduction indicates the remarkable improvement by co-pyrolysis, both economically and environmentally: more than 90% of waste was converted to valuable products while emission was nearly twenty times less than incineration.

3.2. Products Analysis

BET measurements were performed on pyrolytic chars and activated carbons made from both materials. The results are shown in Table 3 together with other waste textile based activated carbon previously reported. The trend in [11, 16] demonstrated that pyrolysis temperature increase tends to reduce surface area, while activation temperature increase gives the opposite effect. This explained why the specific surface area of unactivated chars in this study was quite high, especially the one from fresh rag, which even surpassed some activated carbon. Further activation is, however, not very effective on char from fresh rags due to low temperature, with only 20% gain in specific surface area-very modest against the fiftyfold achieved in [9]. The benefit of low activation temperature is yield: mass lost during activation is negligible in this study, while nearly half of the material was lost during activation in case 8 [9].

The cases of used oily rags were different. Overall pyrolysis rate was not hindered as the solid yield is similar to fresh material, but WLO prevented the exploitation of the porous structure in raw material, making the surface area three times smaller. This is possibly because the residue from WLO blocked the pores on textile fibre after pyrolysis. The problem was solved by activation: specific surface area expanded considerably, even a little higher than activated carbon from fresh rag, thanks to the catalytic effect of metal additive in WLO, while mass lost remained insignificant. At the end, wasted oily rags were converted to activated carbon with a decent surface area, higher than that typical activated carbon achieved via gas activation.

Table 3. Specific surface area of pyrolytic chars and activated carbons, m^2/g

Case	Textile waste stock	Pyrolysis condition	Activation condition	BET surface area, m^2/g	Reference
1	Cotton + PET	500 °C (20 °C /min), nitrogen, 60 min	No activation	793	This study
2			KOH, 550 °C, 90 min	951	
3	Cotton + PET contaminated by WLO	500 °C (20 °C /min), nitrogen, 60 min	No activation	267	
4			KOH, 550 °C, 90 min	1178	
5	Cotton + Polyester	500 °C (10 °C /min), nitrogen, 60 min	No activation	5.216	[6]
6			$ZnCl_2$, with pyrolysis	1605	
7	Flax	800 °C (2 °C/min), nitrogen, 60 min	No activation	30	[9]
8			KOH, 800 °C, 60 min	1564	
9			KOH, 800 °C, 120 min	1656	
10			CO_2, 825 °C, 360 min	867	
11	Acrylic	800 °C (5 °C /min), nitrogen, 120 min	Steam, 800 °C, 180 min	148	[11]
12			Steam, 850 °C, 180 min	354	
13			Steam, 900 °C, 180 min	619	
14		900 °C (5 °C /min), nitrogen, 120 min	Steam, 800 °C, 180 min	52	
15			Steam, 850 °C, 180 min	204	
16			Steam, 900 °C, 180 min	373	
17	Acrylic	700 °C (5 °C/min), nitrogen, 120 min	Steam, 900 °C, 120 min	752	[16]
18	Flax	450 °C (2 °C/min), nitrogen, 60 min	Steam, 800 °C	877	[17]
19	Hemp			776	
20	Jute			840	
21	Coir			822	
22	Abaca			860	

Besides, solids, liquids are also valuable products of pyrolysis. Until the steep rise in global petroleum production in the middle of 20th century, pyroligneous acid from wood distillation was a substantial source for acetic acid, acetone, and methanol, among other chemicals. Nowadays, when these chemicals can be produced from other sources at lower cost, the liquid product is consumed as fuel to avoid the need for purification. When pyrolysis is performed on renewable material like biomass, the liquid product, called bio-oil, is a renewable fuel that can replace traditional ones from fossil sources. For example, pyrolysis of suitable biomass, such as areca nut husk, can produce bio-oil at 49.63% yield and 38.16 MJ/kg higher heating value [18]. Wastes with high heat of combustion and availability are also good raw materials. Pyrolytic oil from waste tires has been commercialized and can be mixed with diesel oil and supplied to diesel engines [19]. To the contrary, waste fabric, especially from natural fibre, has not been a good raw material for liquid biofuel. The reason is that during pyrolysis, cellulose usually decomposes to acetic acid-a corrosive substance with low heat of combustion. Previous pyrolysis studies on cotton textile waste produced bio-oil with 18 – 20 MJ/kg higher heating value [20] or even lower-only 11.5 MJ/kg [21].

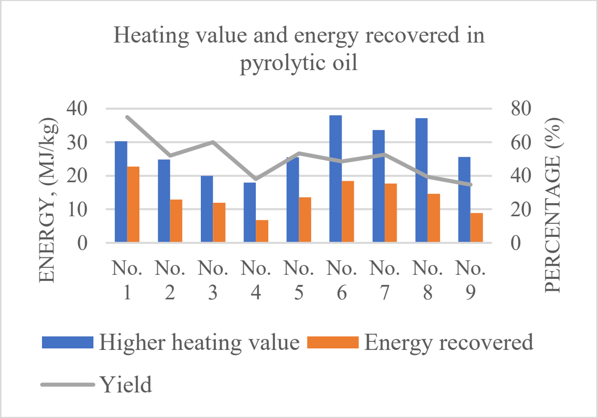

Fig. 3. Pyrolytic oil from different materials

In this study, since WLO intervened with fabric pyrolysis, hindering the formation of small molecules, it is expected that pyrolytic oil may be a more capable fuel. Fig. 3 shows the pyrolytic oil analysis results of this study and previous ones for comparison, the list of reports on pyrolytic oil yield is in Table 4. The effect of material on pyrolytic oil is clear: heating value of pyrolytic oils from cotton, which comprises mostly cellulose, are lowest; lignin adds some heating value as in the case of sugarcane bagasse, walnut shell, or corn stover; components with higher heating value, like tire or vegetable oil in cashew nut shell, raise the heating value further. In most cases energy recovered in pyrolytic oil is around 10 MJ from 1 kg of raw material. Even in case of waste tire, this number is still below 20 MJ-circa 50% heating value of raw material. The heating value of pyrolytic oil from oily rags was 30.3 MJ/kg, which agreed well with a previous study on mixture of PET and biomass [12], and the high yield helped recover 22.73 MJ from 1 kg of the waste. Therefore, regarding the energy aspect, the pyrolytic oil from oily rags is a good candidate for blending and partly replacing conventional fuel for engine or furnace use. Further research on the compatibility with engine or furnace materials is, of course, still required for its adoption on a large scale.

Table 4. Studies on pyrolytic oil from various types of raw material

No.	Stock	Reference
1	Cotton + PET, contaminated by WLO	This study
2	Sugarcane bagasse	[13]
3	Cotton	[20]
4	Cotton	[20]
5	Walnut shell	[22]
6	Waste tire	[22]
7	Walnut shell + waste tire	[22]
8	Cashew nut shell	[23]
9	Corn stover	[24]

The last pyrolysis product effluent gas is planned to be vented like flue gas. The gas composition varied during pyrolysis as illustrated in Fig. 4. Excluding nitrogen, carbon dioxide is the component with the highest concentration, and the second is carbon monoxide. Their concentrations were quite high at the beginning but dropped to nearly zero after 35 minutes. Hydrocarbons appeared at a lower concentration but persisted until the end of pyrolysis. The total amount of hydrocarbons is still less than carbon monoxide. Nitrogen oxides are the least components with a dropped maximum concentration at 301 ppm. The average concentrations of carbon oxides, nitrogen oxides, and hydrocarbons in effluent gas were summarized in Table 5. Thanks to the reduction environment in the pyrolysis reactor, nitrogen oxides level was quite low, conforming to the regulations of flue gas by many countries: Vietnam, China, EU, US,… Although hydrogen fraction was not measured, it had always been less than the carbon monoxide fraction in previous studies on the pyrolysis of spent disposable wipes [25], cashew nut shells [23]. Accordingly, it is safe to assume that the hydrogen value in effluent gas is negligible.

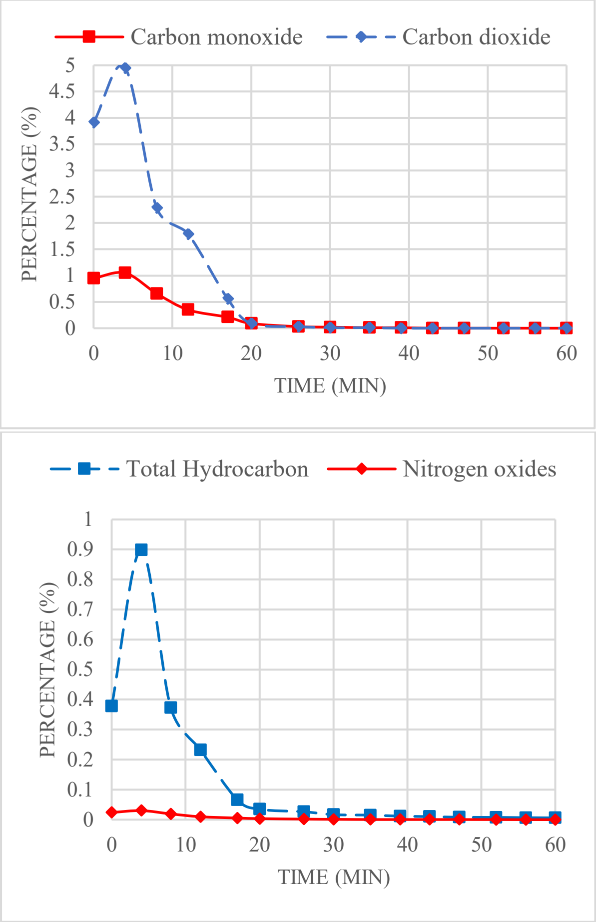

Fig. 4. Effluent gas composition during pyrolysis

Table 5. Average concentrations in effluent gas, ppm

Carbon monoxide	Carbon dioxide	Total hydrocarbon	Nitrogen oxides
1980.8	7953.3	1295.8	57.8

4. Conclusion

Cotton-PET mixed fabric scrap can be reused as wipes or pyrolyzed to produce activated carbon. During pyrolysis, more than half of the fabric is converted to gas and becomes a burden to the atmosphere. Once the fabric is used to wipes and contaminated with WLO, it can also be pyrolyzed to produce char and oil. The surface area of the char is then several times reduced but can be expanded to 1178 m^2/g, even larger than the one from fresh fabric, by additional activation. The presence of WLO shifts pyrolysis product from gas to liquid, resulting in very low gas yield and high liquid yield. Emission from pyrolysis is then substantially reduced as mass and energy were conserved in char and pyrolytic oil. The heating value of the oil is 30.3 MJ/kg, quite high compared to bio-oils. When the oil is combusted, it releases nearly the same energy as burning original fabric but less CO_2 because a large portion of carbon has been fixed in the char. Overall, pyrolysis is an efficient method to convert contaminated oily rags to useful materials and energy.

Acknowledgments

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