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Evaluation of the efficiency of chemical and thermal regeneration of rice husk hybrid adsorbent by glycerol adsorption

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Abstract This study investigates the regeneration efficiency of the chemical and thermal treated activated carbon previously used for biodiesel purification. Some analyses were carried out to find the chemical and physical modifications on the surface of regenerated activated carbon. In order to evaluate the regeneration efficiency, the regenerated activated carbons were used for glycerol removing. Therefore, the efficiency was studied by comparing adsorption rate of glycerol between the fresh and regenerated activated carbons. The analysis showed the similar chemical and physical characteristics between the fresh adsorbents and thermal regenerated adsorbents contrarily to the activated carbon regenerated chemically. The rate glycerol removal was 59.9% for thermally regenerated activated carbon, whereas solvent regeneration activated carbon, it was 46.8%. The regeneration efficiencies of 84% and 107% were found for chemical solvent and thermal regeneration respectively. The thermal regeneration allowed to regenerate the spent activated carbon with better adsorption capacity.

Keywords regeneration efficiency, activated carbon, glycerol, biodiesel, removal efficiency

Nomenclature
AC-f: fresh Activated carbon
AC-r: regenerated Activated carbon
AC-t: thermal regenerated activated carbon
AC-c: chemical regenerated activated carbon
RE: regeneration efficiency
R: removal capacity
C_0: initial concentration
C_e: equilibrium concentration
dTG: differential thermal gravimetric
TGA: thermal-gravimetric analysis

1. Introduction

Activated carbons have widely used in several applications for removal of heavy metal ions [1-2] and organic pollutants [3-4] from water and treatment of pollutants present in liquid gaseous effluents [5-6], and due to their excellent adsorbent properties and large surface areas [7]. But, depending on their adsorption capacity, they

become saturated after some using. Once activated carbon is exhausted, it must be disposed or recycled by using some process such as land filling, incineration and thermal reactivation. Methods used to regenerate the service life of the activated carbon include; biodegradation (metabolism and co-metabolism) [8], chemical regeneration [9], electrochemical regeneration [10], and steam regeneration [11], microwave-assisted pyrolysis regeneration and thermal regeneration [12-15].

Replacement and subsequent disposal of spent granular activated carbon is expensive. By increasing the service life of the granular activated carbon, costs can be decreased [8]. Regeneration of the adsorbent material is of crucial importance in the economic development [16]. The economy of regeneration of the carbon is therefore high and that the regeneration process is a successful way to cut cost of preparation activated carbon [17]. According to Dwivedi *et al.* regeneration renews the active sites without damaging the capacity of the adsorbent, doing it reusable in several adsorptions and desorption cycles. Furthermore, regeneration should also ensure that eluted solution is not posing any disposal problem waste in terms of high acidity [16].

A potential alternative approach is to chemically regenerate activated carbon with an appropriate solution capable to remove organic contaminants, having a range of physico-chemical properties such as ethanol [18]. Some authors' uses hot distilled water to wash the used activated carbon to clear the surface and pores of physically bound materials, with the most minimal chemical interaction. This transformed the spent activated carbon into less toxic by-products and the sorption capacity of the carbon is re-established; thus, increasing the useful life of the activated carbon and the costs of water treatment are reduced [19].

The regeneration methods applied on the spent activated carbon for the purification of biodiesel in our study are the simplest, such as solvent washing or thermal treatment. Ethanol was used for the production of Jatropha *curcas* biodiesel in our study, instead of methanol. Moreover, ethanol is considered as bio solvent because it is obtained by fermentation of polysaccharides or lignocellulosic materials. To follow the reusability of activated carbons, we used the following analytical techniques: methylene blue adsorption, IR, SEM, etc.

Thus, the objective is (1) to characterize the regenerated adsorbents and (2) to study the adsorption capacity of glycerol.

2.Material and methods

2.1Material

The spent activated carbon proceeded from fresh activated carbon AC-f already investigated in our previous work [20]. It was prepared with rice husk as precursor and zinc chloride as chemical activation agent.

2.2Methods

2.2.1Thermal-gravimetric analysis (TGA)

Thermal analysis objects to determine the optimal temperature for regeneration of the exhausted activated carbon. Therefore, the thermogravimetric analyses were was investigated on glycerol, crude oil of Jatropha *curcas* and his biodiesel. These analyses were carried out at a heating rate of 10 °C/ min under gaseous nitrogen, from room temperature to 1000 °C.

2.2.2.Scanning Electronic Microscopic Coupled Energy Dispersive X-Ray Spectroscopy (SEM/EDX)

The device used (JEOL JSM 6460) is equipped with a secondary electron detector allowing a morphological vision and a backscattered electron detector to study the contrast of chemical composition according to the atomic number.

2.2.3.Infrared analysis

Infrared spectra were recorded in a wavelength range ranging from 400 to 4000 cm^{-1} with a resolution of 2 cm^{-1} using a Perkin-Elmer Fourier Transform 1720-x spectrophotometer. The measurements are carried out on KBr pellets obtained by pressing powders in a ratio of 3 mg of activated carbon/1 g of KBr.

2.2.4.Regeneration of activated carbon

The regeneration of spent adsorbent was developed using two type of methods such as solvent washing (chemical) and thermal treatment according the figure 1.

Recycling by solvent washing: in Erlenmeyer flask of 150 mL, 01.0 g of used activated carbon is soaked in 10 mL of absolute ethanol. The mixture was then shaking at 150 rpm maintaining the temperature at 50 °C. The

washing process was repeated at five times and the activated carbon recuperated by filtration was dried at 100 °C in oven for 01 hour and is named AC-t.

Recycling by heat treatment: 01.0 gram of activated carbon previously used for the adsorption of glycerol is introduced in the furnace to be carbonized at 400 °C for 01 hour with a temperature rise rate of 30 °C/min. The regenerated activated carbon was washed with distilled water several times and dried in oven at 105 °C for 04 hours and also named AC-c.

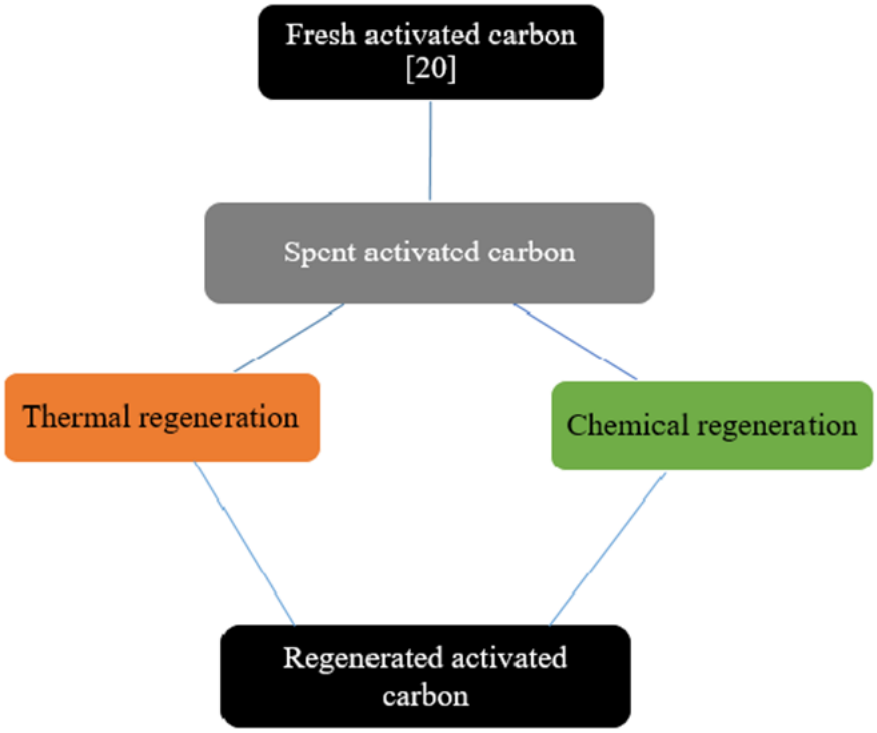


Figure 1: Methods of the regeneration processes

2.2.5.Adsorption of methylene blue

Methylene blue sorption study was conducted in a routine manner by batch technique. For batch experiment a fixed amount (1.00 gm) of recycled activated carbon (thermal or solvent regeneration) was placed into 250 mL flasks containing 100 mL of methylene blue aqueous solution at 200 mg/L. The system was agitated using a platform shaker at 150 rpm for a predetermined time until the adsorption process reached equilibrium. The residual concentration at equilibrium was analysed by UV-visible spectrophotometry at 665 nm.

2.2.6.Adsorption experiments

This parameter has been investigated on glycerol adsorption by activated carbon. The efficiency capacity of regenerated carbon was performed with 4% (w/w) activated carbon/biodiesel, heated at 50 °C under constant stirring 200 rpm for 30 minutes. Indeed, regenerated activated carbon was placed in the flask of 50 mL containing 10.00 g of biodiesel. Each adsorption test was carried out in triplicate to give average. After separation by centrifugation, the residual concentration of glycerol in the biodiesel was determined by the UV-visible method described by Bondioli et *al*. [21]. The percentage removal efficiency R (%) for each activated carbon was calculated using the following equation:

$$R\ (\%) = \frac{C_0 - C_e}{C_0} \times 100$$

Where C_0 and C_e are initial and equilibrium phenol concentrations, respectively (mg/l).

2.2.6.Calculation of regeneration efficiency

The regeneration efficiency (RE) was defined as the proportion of adsorption capacity of the regenerated activated carbon to the adsorption capacity in the fresh activated carbon:

$RE = (R_{AC\text{-}r} / R_{AC\text{-}f}) \times 100$

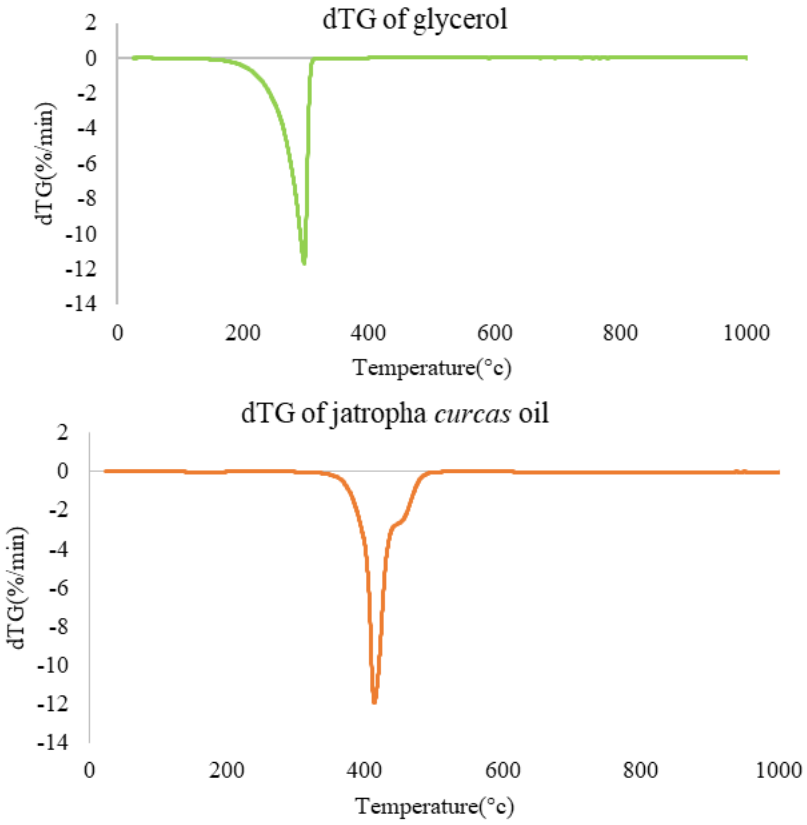
As $R_{AC\text{-}r}$ is the regeneration efficiency of fresh activated carbon and $R_{AC\text{-}r}$ is the regeneration efficiency of regenerated activated carbon.

3.Results and discussion

3.1.Thermal analysis

The dTG curves of glycerol, crude oil of Jatropha *curcas* and biodiesel were showed in figure 2. The dTG curve of glycerol exhibited the mass loss between 150 and 350 °C. The crude oil of Jatropha revealed a main degradation peak around 300 °C and 500 °C. These results were agreed with Kenda et *al*. who have reported thermal decomposition of triglycerides from Jatropha oil and its partial glycerides between 250 and 475 °C [22]. The dTG curve of biodiesel showed two weight losses within temperatures range of 150-380 °C and 380-500 °C. The first one was noticeable by a high mass loss due to the volatile and semi-volatile compounds [9] and the second was owed to the decomposition of residual biodiesel on spent activated carbon.

A comparative analysis of these three curves showed that 500 °C was an adequate temperature to decompose all the organic matter adsorbed on the surface of the activated carbon. The study revealed that activated carbon obtained at 400 °C (AC) is the best performing material. In order to stay in the limit of the preparation temperature of AC and to avoid structural and functional modifications, the best regeneration temperature was 400 °C. After the first use, the AC was separated from biodiesel by filtration and regenerated at the suitable temperature revealed by thermal study 400 °C.



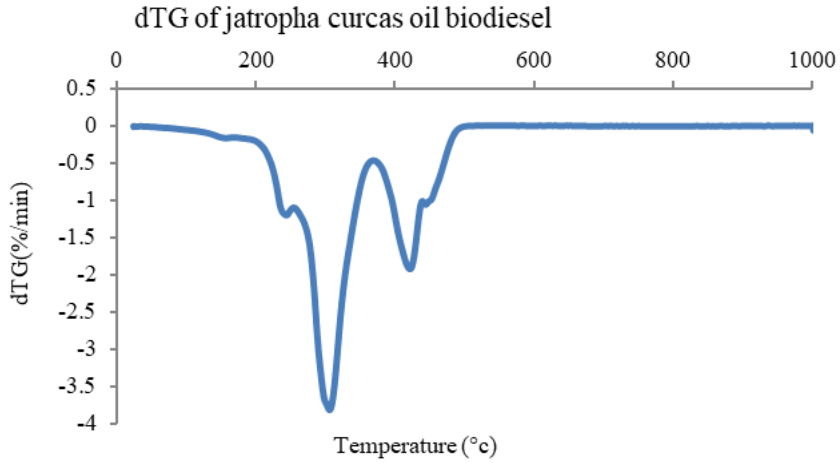


Figure 2: dTG curves of glycerol, Jatropha curcas oil and biodiesel

3.2.Characterization of recycled adsorbent materials

The methylene blue adsorption of the activated carbon AC-t was evaluated and compared to the fresh activated carbon AC and the results were plotted on the figure 3. The maximum adsorption capacity was 21.2 mg / g for the activated carbon AC-t. This value represented 50% comparatively to the maximum adsorption capacity of the fresh activated carbon AC-f (43.9 mg/g). According to the results, the regeneration of activated carbon by solvent washing did not allow to restore the adsorption capacity in the second cycle.

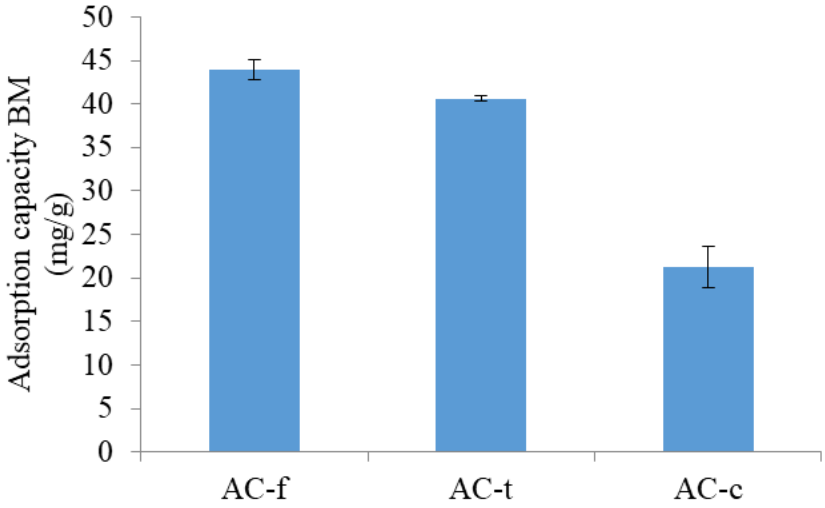


Figure 3: Methylene blue (MB) adsorption capacity

Contrarily to the solvent regeneration, the activated carbon regenerated by thermal way gave an appreciable adsorption of methylene blue. Maximum capacity is 40.6 mg/g for AC-c against 43.9 mg/g for AC-f. These results prove that blocking or filling of pore would be responsible to the decrease of glycerol adsorption capacity and possibly other compounds such as ethyl esters on the surface of the catalysts.

Regeneration of activated carbon under wet conditions is not efficient because during the cooling phase, re-adsorption of water molecules could occur [14]. The authors of this study recommend a high temperature regeneration of short duration. Other studies have shown that the regeneration temperature may be higher than that of our study (400 °C). Indeed, these authors recommend that the adsorbents can be treated at high temperature and by reductive methods. Therefore, the adsorbents are carbonized hydrogen atmosphere at 400-500 °C [23-24].

3.3.Surface chemical functions of activated carbons

The FTIR spectra of AC-f, AC-c and AC-t recorded between 400 and 4000 cm^{-1} are presented in figure 5. The infrared curve of AC-t shows a new absorption band at 3778 cm^{-1}. This weak absorption band would be attributable to the vibration to O−H bond of the glycerol adsorbed on activated carbon after the first cycle. This result suggests that regeneration with ethanol does not completely remove pollutants. Comparatively to the fresh activated carbon, the peaks intensities of different functions decrease due to the leaching by ethanol.

Through AC-c FTIR spectrum, there are no additional absorption bands compared to original activated carbon. The temperature of 400 °C is sufficient to decompose the organic material adsorbed on the activated carbon surface. These results confirm the results obtained on the methylene blue indices.

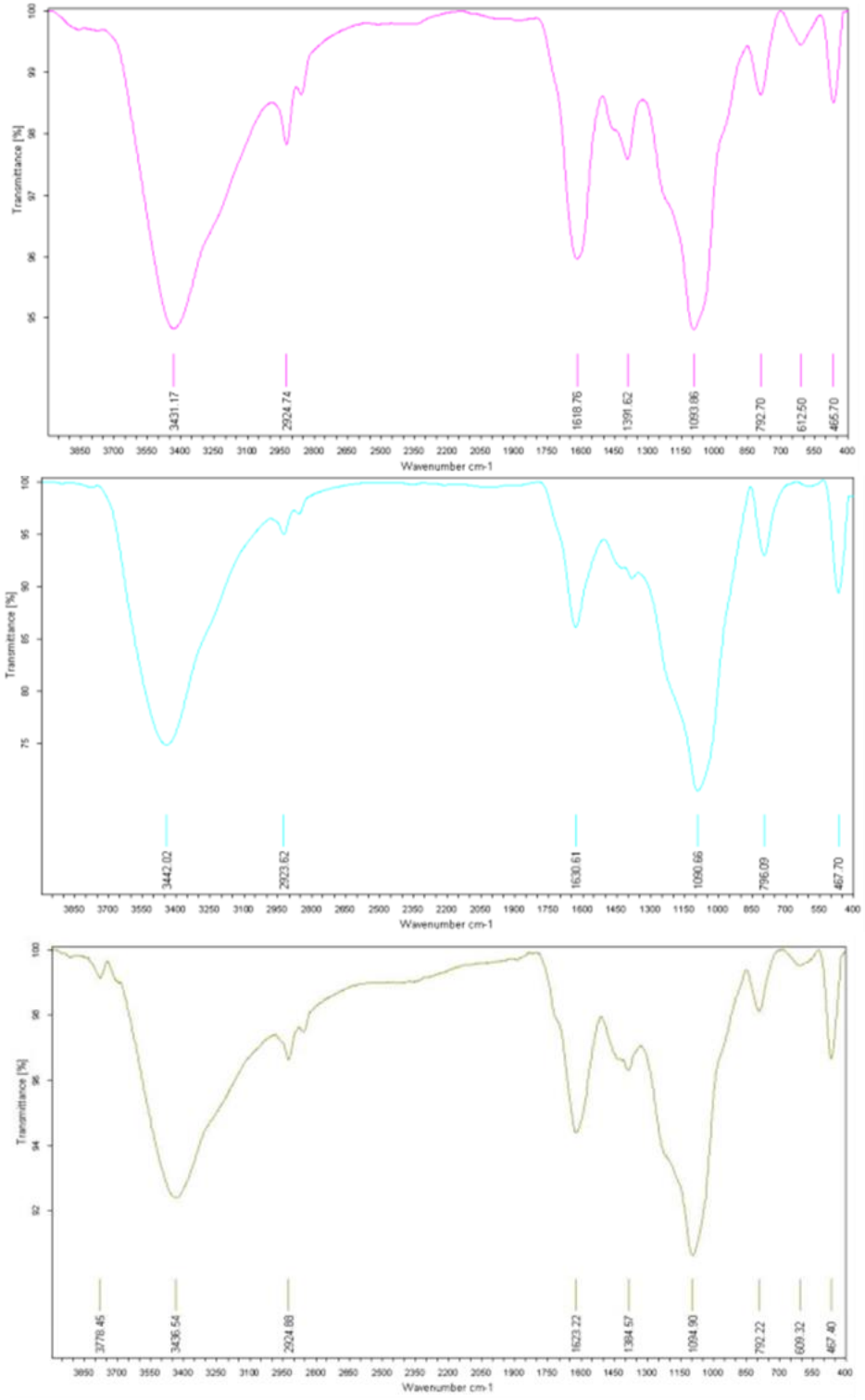


Figure 4: Infrared spectra (A): AC-f, (B) AC-c and (C) AC-t

3.4.Scanning Electron Microscopy analysis

The SEM of the spent and the regenerated activated carbon are shown in Figure 4. It was taken to observe the surface characters of AC before and after regeneration. It was clear that the surface of the AC-s (**C**) was covered by pollutants. After regeneration, impurities were removed from the surface of the AC-t (**B**) was visible and his morphology was comparable with fresh activated carbon (**A**).

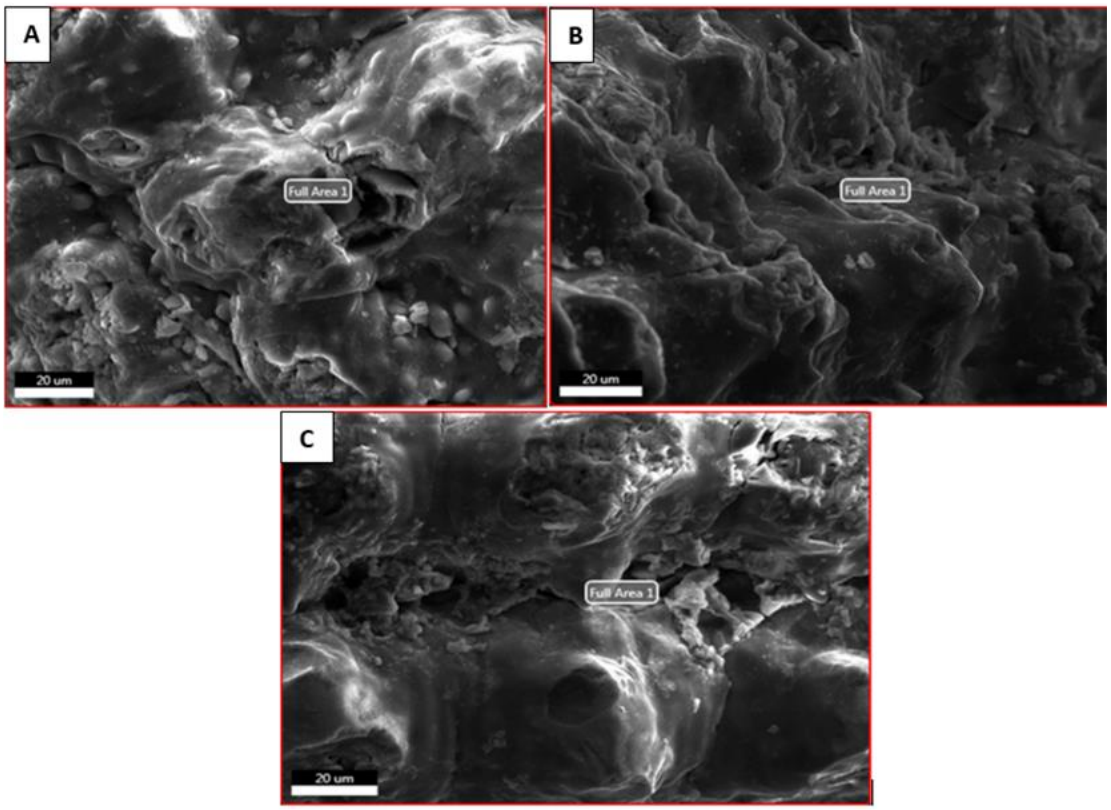


Figure 5: Scanning Electronic Microscopic of spent and regenerated carbons; (A): AC-s, (B): AC-t and (C): AC-c

3.5.Energy Dispersion X-Ray Spectroscopy

Table 1 below gave the chemical composition of fresh, thermal and wet recycling activated carbon. The presence of carbon element in this analysis attests clearly to the presence of carbon in the sample. The carbon and oxygen contents increased significantly in the activated carbon after wet regeneration. Although glycerol is a small molecule, it provides three carbon and oxygen atoms per molecule. The increase of the content for these two atoms reveals the presence of glycerol on the washed activated carbon as reported on the infrared spectrum. The silica content of the recycled carbon has decreased comparatively to the fresh activated carbon due to the increase carbon and oxygen elements.

Contrarily to wet regeneration, the carbon content obtained by thermal recycling is almost identical to that of the initial activated carbon. Therefore, this result confirms the FTIR analysis which presented similar spectra of regenerated and initial carbon. The carbonization temperature at 400 °C is effective temperature for the activated carbon regeneration used for the glycerol adsorption. This activated carbon has a higher glycerol adsorption rate than the initial carbon. This result confirms that the activated carbon recycled after use has been freed from glycerol compared to that recycled by washing with ethanol.

Table 1: Chemical composition by EDX analysis of carbon AC-f, AC-t and AC-c

Adsorbent	Atomic concentration (%)			
	C	O	Si	Zn

AC-f	31,68	51,71	15,30	0,77
AC-c	33	52,76	13,10	0,38
AC-t	31,39	49,23	17,79	1

3.6.Evaluation of the efficiency of regenerated activated carbon

The efficiency of recycled activated carbon on glycerol adsorption was evaluated and the results are shown in Figure 6. The results show that thermal recycling can restore the activated carbon capacity more than wet recycling. Indeed, the thermal and chemical regeneration give the percentage of glycerol adsorption of 59.9% and 46.8% respectively against 55.7% for the initial activated carbon. From the adsorption of glycerol, it is appeared clearly that the activated carbon regenerated by the thermal way showed better regeneration efficiency. The RE (%) reached more one hundred percentage and superior than the fresh activated carbon. The thermal regeneration increased the adsorption capacity of the activated carbon.

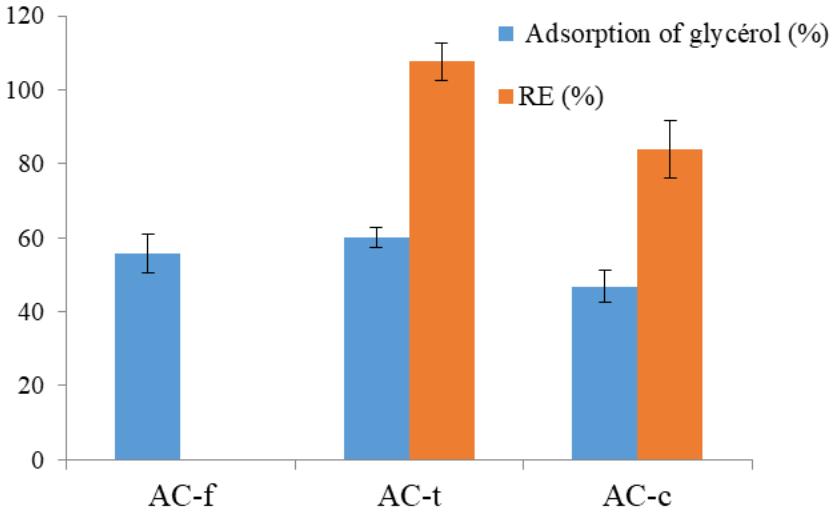


Figure 6: Adsorption of glycerol and regeneration efficiency of regenerated activated carbons
Conditions: mass of biodiesel (glycerol 0.4%) 10 g; mass of activated carbon (0.4 g); time 30 min; speed 150 rpm; temperature 50 °C

3.7.Discussion on efficiency of reused activated carbon

Recycling by heat treatment could decompose all organic constituents adsorbed on the activated carbon, creating new pores, increasing their adsorption efficiency. According to Lashaki et *al*., increasing the regeneration temperature between 288-400 °C, increases desorption intrenched solutes in contracted micropores and mesopores [25]. When the difference between solute boiling point and regeneration temperature is high, the desorption process becomes rapid and efficient. This observation mentioned by these authors could explain the efficiency of activated carbon recycled by carbonization because the boiling point of glycerol is 290 °C and the regeneration is carried out at 400 °C. Also, the value of 400 °C taken as the thermal regeneration temperature is considered sufficient for the complete degradation of the glycerol adsorbed on the activated carbons with regard to the dTG thermogravimetric of the glycerol observed on figure 1. Thermal regeneration eliminated by combustion the organic compounds inserted in the pores and then removed from the surface of the adsorbents.

For the regeneration by washing, the choice fell on ethanol as the solvent for recycling the activated carbon for two reasons. Firstly, glycerol is well soluble in ethanol and secondly, ethanol is a bio-available product. The three hydroxyl groups of glycerol confer this easier dissolution property in ethanol which is also a polar solvent. Ethanol presents as suitable solvent for organic solutes desorption because according to Ghasemzadeh et *al*., it did not have much effect on the regeneration process if the amount organic adsorbates content is low [9].

One study showed the efficiency of recycling for five cycles, and the authors concluded that regeneration does not affect the adsorption capacity of the carbon and that it restores this capacity of the carbon to its initial value [11].

Regarding to our study, five uses are necessary to reach the glycerol concentration of 0.02% according to standards value, with an initial concentration of 0.40%.

In fact, the recycling allowed desorption of the glycerol, possibly the partial glycerides, the free fatty acids and the residual catalysts. Compared to the results of literature, the recycling of the ash from rice husk by absolute methanol (10x50 mL) was still effective after five re-uses in the adsorption of glycerol [26] with an adsorption rate of 55%. According to studies reported on the glycerol removal, and partial glycerides in small proportions, regeneration by washing with methanol is also effective [27]. Similarly, Saengprachum et *al*. have also used methanol to regenerate spent activated carbon have shown that regeneration can be carried out up to five cycles [28].

The effect methanol washing was also confirmed by Wall et *al*. which have shown the rate of flow of methanol during regeneration to have a significant effect on washing efficiency [29]. The glycerol content in biodiesel has been reduced from 0.08% to 0.02% with the recycled resin (T45BD). The conventional washing method can also use ethanol as a solvent for desorbing glycerol from the adsorbent. In fact, washing the Purolite PD 206 resin with ethanol at 25 °C allows the glycerol to be discharged after 300 min. This explains why the recycling process remains long under low temperature conditions [30].

The thermal treatment gave the better regeneration efficiency comparatively to the chemical regeneration. From an economic point of view and environmental respect, the result can be discussed. Indeed, the chemical regeneration in our study used bioethanol. This process is simple to apply and consumes less energy. According to Kulkarni et *al.*, Solvent washing was very effective and advantageous from environmental point of view [23]. Hence, by comparing the experimental temperatures that were applied in this study and the TGA results, chemical regeneration was more effective than thermal regeneration, as thermal regeneration caused high energy consumption which led to high costs as well as carbon loss [9].

4.Conclusion

The regeneration of spent activated carbons after glycerol adsorption from biodiesel, was performed by solvent washing and heat treatment. Methylene blue adsorption characterization showed that thermally recycled activated carbons adsorbed methylene blue more than those recycled by solvent washing. Thermal regeneration is therefore more efficient than solvent washing. Recycling the spent activated carbon thermally removes glycerol and other components and increase in carbon and oxygen in the activated carbon regenerated by simple washing. This constitutes an indication of the presence of non-eliminated glycerol. In fact, thermal recycling maintains the porosity of activated carbon by increasing the adsorption capacity of methylene blue. This result was confirmed with the rate of glycerol adsorption. The activated carbon regenerated by the heat treatment gives a glycerol adsorption rate slightly higher than that of the initial activated carbon, ie 59.9% against 55.7%. Thus, AC-t presents a better result with regeneration efficiency 107% instead AC-c showed 84%.

It is therefore possible, in the application of the results, to consider the purification of the same sample of biodiesel with a glycerol content of 0.4% m / m at the start by the same activated carbon from rice hulls recycled 4 times for a total of 5 uses in order to reach the rate of 0.02% of glycerol accepted according to the quality standard for biodiesel.

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