

# Microwave Regeneration of Swiss Blue Dye loaded Conventional Prepared Activated Carbon from Brewer's Spent Grain.

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**Abstract**—This work has successfully studied microwave regeneration of Swiss blue dye loaded conventionally prepared activated carbon from brewer's spent grain. The objective of the work was production of activated carbon from brewer's spent grain, saturation with different concentrations of Swiss blue dye and its regeneration using microwave technique. Effect of microwave power levels and radiation time on the adsorptive capacity of the regenerated carbon was studied with and without an oxidant (10% $H_2O_2$ ) for one cycle. Effect of six adsorption-regeneration cycles on the adsorptive capacity and regeneration efficiency of the carbon was studied. The results showed that increase in power levels from 18% to 81% increased the adsorptive capacity of the regenerated carbon from 119.36mg/g to 122.07mg/g, further increase to 100% decreased adsorptive capacity to 118.48mg/g. Increase in radiation time from 5mins to 10mins increased the adsorptive capacity from 118.22mg/g to 122.07mg/g, while further increase to 20mins decreased adsorptive capacity to 118.36mg/g. Increase in number of cycles from zero to two increased the adsorptive capacity from 188.947mg/g to 122.074mg/g, and regeneration efficiency from 100% to 102.63%, while further increase to six cycles decreased the adsorptive capacity to 118.836mg/g, and regeneration efficiency to 99.27%. FTIR analysis of the regenerated carbon showed fewer peaks compared to the raw carbon. The properties of the regenerated carbons studied showed that microwave regeneration preserves the porous structures of the activated carbon. It is concluded that microwave heating technique can effectively regenerates spent activated carbon.

**Keywords**—*Microwave, Regeneration, Saturation, Spent grain, Swiss blue dye*

## I. INTRODUCTION

There are economic and environmental incentive to regenerate products derived from waste treatment processes. One such product is granular activated carbon, a solid adsorbent used to remove a wide range of organic compound from industrial and municipal wastewater [1]. The relative high initial cost of activated carbon, and the fact that disposal of spent adsorbent via landfills or incineration adds pollutant to the environment, mandates serious consideration of carbon regeneration.

Current available method for regeneration include; chemical, biological, oxidation, ultrasound, and photo catalytic method [2], [3], [4], [5], [6]. Among them heating regeneration technology was most widely used, but it has many disadvantages such as long regeneration process, high energy input, and low quality products. The use of microwave can mitigate the defects in the process of heating regeneration and reduce the energy needed for regeneration [2]. The most common efficient regeneration technique is microwave regeneration method [7]. By employing microwave energy, the regeneration time can be considerably shortened compared to conventional thermal heating [8]. Microwave heating preserved the porous structure of the regenerated activated carbon more efficiently than treatment with conventional device [9].

Brewers' grains often referred to as spent grains are major co-products of the brewing process, generated at a rate of up to 30% of the weight of the initial malt grist [10]. Worldwide generation of BSG has been estimated at 30 billion kilograms per annum. Traditionally this material has either been discarded or sold as animal feed, but increasingly industry is seeking to find added value applications which change traditional views of waste streams and reclassify them as co-products. In this work, activated carbon was prepared from brewers' spent grain via conventional activation method.

Swiss blue dye is a basic dye that is typically cationic or positively charged and reacts well with a material that is anionic or negatively charged. Swiss blue dye

was chosen in this study because of its known strong adsorption onto solids.

## II. METHODOLOGY

### a) Preparation of Activated Carbon

The method used for the preparation of activated carbon was in accordance with the work done by Hammed [11] with slight modification.

The sample was washed exhaustively with deionized water to remove adhering dirt particles from the surface. Dried sample was ground and screened to the desired mesh size of 1 – 2mm. The carbonization process was performed by loading a known quantity of the precursor into a vertical furnace, and the temperature was ramped from room temperature to the desired temperature of 800°C. The char produced was soaked in 6M KOH solution with 1.5:1 impregnation ratio defined as the volume of activating agent to weight of char (KOH: Char). The mixture was then dehydrated in an oven for one hour to remove moisture.

For the conventional activation, it was done under the same condition as carbonization but to a different final temperature of 850°C and time of one hour. The resultant activated carbon was washed repeatedly with 0.1M HCl and distilled water until PH 6-7 was reached in the residual liquid.

### b) Experimental set- up of microwave oven with the modifications.

This work utilized a modified Sonik domestic microwave oven model SMW-90023 Japan, with a maximum power output of 900W delivered at a frequency of 2450MHz. As part of the modifications, the microwave casing was penetrated from the top to provide fittings to support the quartz glassware reactor. The reactor was opened from the top end to allow for the escape of the pyrolysis gases. The upper surface of the oven had a removable cover connected to a stainless steel pipe from which the volatiles exit. The volatiles were then transferred to a condensing system made up of large absorbing bottle. The quartz glassware which was mounted inside the microwave cavity consisted of 100mls round bottom flask into which the samples was loaded, and a quartz quick fit connection collar which joined the flask with the outlet connection. Quartz was selected as reactor of choice because is a good material to be used in microwave reactors, it resist temperatures, transparent to microwaves and can resist thermal shock much better than traditional laboratory glass such as pyrex [12]. However, the reactor was not completely invulnerable and was found to show signs of devitrification after extended use. Thus, periodic replacement of the reactor was done.

### c) Characterisation of activated carbon

Fourier transformed Infrared Spectroscopy (FTIR)

The surface functional groups and structure were studied by fourier transform infrared spectroscopy [Buck 530 IR] England. The FTIR spectra of the raw material and activated carbon were scanned at a wavelength of 600–4000nm.

### Determination of Surface acidity

The surface acidity was done according to the work done by Foo and Hameed [13]. The surface acidity was estimated by mixing 0.2g of activated carbons with 25mls of 0.05M NaOH in a closed flask, and agitated for 48hrs at room temperature. The suspension was decanted and the remaining NaOH was titrated with 0.05M HCl.

### Determination of Surface basicity

The surface basicity was measured according to work done by Foo and Hameed [13]. This was measured by titrating with 0.05M NaOH, after incubation of 0.2g of activated carbons with 0.05M HCl.

### Determination of pH of Point of Zero Charge (pHpzc)

The pH of point of zero charge (pHpzc) of the sample was determined by the solid addition method. To a series of five 100ml conical flasks, 45ml of 0.01mol/dm<sup>3</sup> of KNO<sub>3</sub> solution was transferred. The initial solution pH values (pH<sub>i</sub>) were roughly adjusted from pH 2 to 10 by adding either 0.01mol/dm<sup>3</sup> HCl or NaOH using a pH meter. The total volume of the solution in each flask was then made up to 50ml by adding KNO<sub>3</sub> solution which was accurately noted, and 0.10g of the sample was added to each of the flask, which was then securely capped immediately. The suspension were then manually shaken and allowed to equilibrate for 48 hours with intermittent manual shaking. The final pH values (pH<sub>f</sub>) of the supernant liquid were then noted. The difference between the initial and final pH value was plotted against the pH<sub>i</sub>. The point of intersection of the resulting curve at which  $\Delta\text{pH} = 0$  gives the pHpzc.

### Determination of pH

The standard test method for determination of activated carbon pH according to Ekpette and Horsfal,[14] was used. 1g of the activated carbon was weighed and transferred into a beaker. 100ml of distilled water was measured and added and stirred for one hour. The sample was allowed to stabilize before pH was measured using a pH meter.

### Determination of moisture content

Thermal drying method was used in the determination of moisture content of the samples according to Rengaraj [15]. 1.0g of the dried activated carbons were weighed in triplicates and

placed in washed, dried and weighed crucible. The crucibles were placed in an oven and dried at 105°C to constant weight for 4 hours.

The percentage moisture content (%MC) was computed as follows:

$$\text{Moisture \%} = \frac{\text{Loss in weight on drying}}{\text{Initial sample weight}} \times \frac{100}{1} \quad (1)$$

Determination of the amount of iodine adsorbed

This method was according to work done by Gimba and Musa [16]. A stock solution was prepared (0.005mol/L) containing 2.7g of iodine crystal and 4.1g of potassium iodine per litre. The prepared stock solution was standardized using a standard solution of sodium thiosulphate. Into a 100cm<sup>3</sup> volumetric flask, 0.5g of the activated carbon and 10cm<sup>3</sup> of 5% V/V hydrochloric acid was introduced.

The flask was swirled until the carbon was wetted. Then 100cm<sup>3</sup> of the stock iodine solution was added and agitated at a fast speed using an electric shaker for a period of 60 minutes. The mixtures were filtered through a sintered glass crucible. An aliquot portion (20ml) was titrated with 0.1M sodium thiosulphate using starch as indicator.

The concentration of iodine adsorbed by the activated carbon at room temperature was calculated as amount of iodine adsorbed in milligrams.

$$\frac{\text{Img}}{\text{g}} = \frac{B-S}{B} \frac{V.M}{W} \times 253.81 \quad (2)$$

Where B and S are the volume of thiosulphate solution required for blank and sample titration respectively. W is the mass of activated carbon sample. M is the concentration (mol) of the iodine solute. 253.81 is the atomic mass of iodine and v is 20ml aliquot.

Volatile matter

The procedure was according to Onwu [17]. This is the loss of weight converted for moisture when a sample is heated in a specified apparatus under specified condition. 1g of moisture free sample was put into a furnace at 1200K for a period of 7 minutes. The sample was kept covered and cooled rapidly to prevent oxidation (the aim was to remove only the VM without affecting the fixed carbon).

The loss in weight is measured.

$$\%VM = \frac{\text{Initial wt}-\text{final wt}}{\text{initia weight}} \times \frac{100}{1} \quad (3)$$

Ash Content

This is the powdered residues left after combustion. The platinum crucible was weighed. 1gm of the sample was taken in the crucible and weighed. The sample was kept in a muffle furnace for 3hours at temperature of 650°C. This was taken out and kept in desiccators to cool down, the weight was taken. It

was taken back to furnace for 30 minutes to establish that constant weight was obtained.

$$A(\%) = \frac{F-G}{B-G} \times 100 \quad (4)$$

Where: G = mass of empty platinum crucible

B is the mass of crucible plus sample, F is the mass of crucible + ashed sample

Fixed Carbon Content

Fixed carbon was calculated by difference.

$$\% \text{ carbon} = 100 (\%VM + \%moisture + \% \text{ Ash}) \quad (5)$$

The Bulk Density

For the determination of the bulk densities, a method of Azubike et. al [18] was used. 25g of the samples were accurately weighed and poured into a 100ml graduated cylinders each. The cylinder was stopped and the bulk volume (V<sub>o</sub>) was recorded.

$$D_{\text{bulk}} = \frac{W}{V_o} \quad (6)$$

Where W was the weight of the sample, and V<sub>o</sub> was the volumes of the bulk samples.

Determination of Surface Area

Surface area determination was according to Sears' [19]. 1.5g of the activated carbon was agitated in 100ml dilute HCl at pH of 3. Then, 30g of sodium chloride was added while stirring the suspension; the volume was made up to 150ml with deionised water. The solution was titrated with 0.1N NaOH to raise the pH from 4 to 9 and the volume (V) recorded. The surface area was calculated from sears' equation.

$$S \left( \frac{m^2}{g} \right) = 32V - 25 \quad (7)$$

d) Microwave regeneration of Swiss blue dye loaded activated carbons

Microwave regeneration was performed in a modified Sonik domestic microwave oven at a heating efficiency of 2450MHz with a maximum power output of 900W in a Pyrex glass reactor fixed in the chamber of the microwave oven. Initially, the activated carbons were saturated with 100mg/l, 200mg/l, 300mg/l, and 400mg/l of Swiss blue dye for 24 hours. 0.2g of the saturated activated carbon was regenerated at 81% power levels for 10mins. After regeneration, the sample was immersed in a calculated volume of an oxidant (30% H<sub>2</sub>O<sub>2</sub>) at a ratio of (0.02g: 25ml) for one hour at room temperature. It was washed with distilled water and dried. After regeneration, the regenerated carbons were subjected to adsorption process using equivalent initial dye concentrations. The adsorption – regeneration process was repeated for six cycles. The adsorptive capacities were calculated with equation 8, while the regeneration efficiency was calculated using equation 9.

$$q_e = \left( \frac{C_o - C_e}{W} \right) V \quad (8)$$

$$RE(\%) = \frac{q_e \text{ of regenerated AC}}{q_e \text{ of fresh AC}} \times 100 \quad (9)$$

Where  $q_e$  is the adsorptive capacity,  $C_0$  is the initial Swiss blue dye concentration,  $C_e$  is the equilibrium concentration of Swiss blue dye,  $w$  is the weight of the activated carbon,  $V$  is the volume of Swiss blue dye solution.

e) Effect of Microwave regeneration conditions on the Adsorptive Capacity of Regenerated Activated Carbon

Effect of microwave power levels was done at 18%, 36%, 81%, and 100% for 10minutes, while effect of microwave radiation time was studied at 5mins, 10mins, 15mins and 20mins using 81% power level. The activated carbon used was saturated with 100mg/l of Swiss blue dye concentration for 24hours. 0.2g of the saturated activated carbons was desorbed at different power levels for 10minutes and different time intervals using 81% power level. For regeneration with an oxidant, at the end of the time intervals, the sample was immersed in calculated volume of an oxidant (30%  $H_2O_2$ ) at a ratio of (0.02g: 25ml) for one hour at room temperature. It was washed with distilled water and dried. After regeneration, the regenerated activated carbons were subjected to adsorption process with the same 100mg/l of the Swiss blue dye for 3hours. The adsorption was done with activated carbon regenerated with oxidant and that regenerated without the oxidant. This was done to evaluate the effect of the oxidant on the adsorptive capacities of the regenerated activated carbons. The regeneration - adsorption studies was done for one cycle only. The interval with O corresponds to the adsorptive capacity of the fresh activated carbon. The adsorptive capacity of the regenerated activated carbon was calculated with equation 8.

### III. RESULTS AND DISCUSSIONS

a) Effect of Microwave Regeneration Conditions on the Adsorptive Capacity of Regenerated Activated Carbon

Different process factors that affect regeneration efficiency of the spent activated carbon were studied. The desorption-adsorption studies were done for one cycle with cycle O corresponding to the adsorptive capacity of the original activated carbon. The process conditions studied were microwave power levels (18, 36, 81, 100%), and irradiation time (5, 10, 15, 20mins). These process conditions were studied with and without an oxidant. Oxidant that was used in this study is 30%  $H_2O_2$ . 30%  $H_2O_2$  was selected in this work because it is cheap, effective, relatively harmless and highly stable oxidant. Oxidants have oxidizing power to open the block pores and remove the surface functional groups of the spent activated carbons which may block the adsorption of colour [20]. The regenerated activated carbons were soaked in 30%  $H_2O_2$  at room temperature for one hour after

which they were filtered and washed before the next adsorption process.

b) Effect of Microwave Power Levels on the Adsorptive Capacity of Regenerated Activated Carbon.

Microwave power level was considered a crucial variable in examining the impact of irradiation on activated carbon. To better understand this, the saturated activated carbon was exposed to various microwave power levels at 18%, 36%, 81% and 100%. This microwave gives out maximum power of 900W. Therefore, 18%, 36%, 81% and 100% corresponded to 162W, 324W, 729W and 900W respectively. Other conditions were constant at 10mins irradiation time and 100mg/l of initial Swiss blue dye used for saturation. Figure 1 shows the plot of power levels (%) against adsorptive capacity of the regenerated activated carbon. It was observed that the adsorptive capacities of the regenerated activated carbons at different power levels were very close to the fresh adsorbents, though less than that, except 81% that had adsorptive capacity slightly more than the fresh adsorbents. It was evident from the results that increase in power levels enhanced the removal of adsorbed dye due to rapid increase in adsorbent bed temperature. At power level of 81%, it was observed that the adsorptive capacity of spent activated carbon was restored and slightly increased. The phenomenon might be attributed to the reorganization of internal structures as a consequence of the large amount of adsorbate molecule evolving during the microwave irradiation stage thereby activating the activated carbon more [23]. The adsorptive capacities of the adsorbents regenerated without oxidant were less than those regenerated with oxidant. According to Foo and Hammed [13], gradual blockage of porous structure seemed to occur due to the fraction of adsorbed molecules which did not evolve from the carbon surface during regeneration. These molecules may remain inside the pore network of the activated carbons and give rise to carbonaceous residue [24]. This carbonaceous residue blocked the pores thereby decreasing the adsorptive capacities without oxidant. The oxidant tends to clear the pores of the regenerated activated carbon. This finding was in agreement of the work done by Ania et al [24] on the effect of microwave assisted regeneration of activated carbons loaded with pharmaceuticals. They found out that microwave irradiation causes no damage to the carbon but preserve the porous structure and increased the surface area. Equally, Foo and Hameed [13] reported that integration of microwave irradiation promoted the preservation of porosity and adsorption performance in a short heating period. It was observed that excessive power levels beyond 81% reduced the adsorptive capacity of adsorbents. This could be caused by some carbon ablation under a high level of radiation which might undermine the pore structures

[25]. A sudden and intense bright sparks was observed at high power levels resulting in hot spots and red carbons. The whole activated carbons turned red within a few minutes and remained in that condition for the entire time interval. This possibly led

to carbon attrition that reduced the surface area for adsorption. These results were in agreement with my previous studies on the use of microwave irradiation in producing activated carbons.

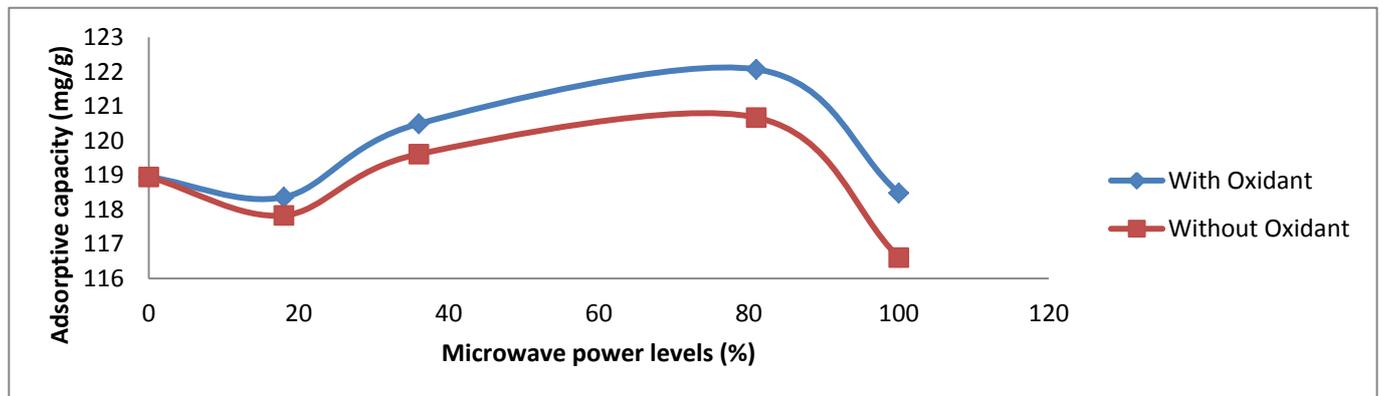


Figure 1. Effect of Microwave power level on the adsorptive capacity of regenerated carbon

c) Effect of Microwave Radiation time on the adsorptive capacity of the regenerated activated carbon.

Radiation time was equally considered as an important factor that affects the regeneration efficiency of an adsorbent. The effect of radiation time was studied at time intervals of 5mins, 10mins, 15mins and 20mins. The time 0 used showed the adsorptive capacity of the fresh adsorbent. The adsorptive capacity was evaluated for one regeneration cycle only. The irradiation time was studied at 81% power level and initial dye concentration of 100mg/l. Figure 2 shows the graph of radiation time versus adsorption capacity of the regenerated activated carbon. It was observed from the plot that adsorptive capacity of the activated carbon slightly decreased as radiation time was increased to 10mins beyond which there was slight decrease again in adsorptive capacity. It was observed that the adsorptive capacity at 10mins was higher than that of the fresh

adsorbent. This trend was observed on the activated carbons regenerated with and without oxidant. The high adsorptive capacity recorded at higher time of 10mins implied that prolonged exposure time promoted an acceleration of temperature which in turn increased the adsorbent bed temperature. The high temperature succeeded in breaking the bonds holding the adsorbate to the adsorbent and further reactivates the carbon. The drop observed beyond 10mins was probably due to sintering effect which largely destroyed the pore walls between adjacent pores. The high temperature can equally lead to collapse of carbon active sites which in turn reduces the adsorptive capacity.

d) Microwave Regeneration Cycles of Swiss blue dye loaded activated carbon.

The activated carbon was saturated with 100mg/l, 200mg/l, 300mg/l and 400mg/l initial Swiss blue dye concentrations for twenty four hours.

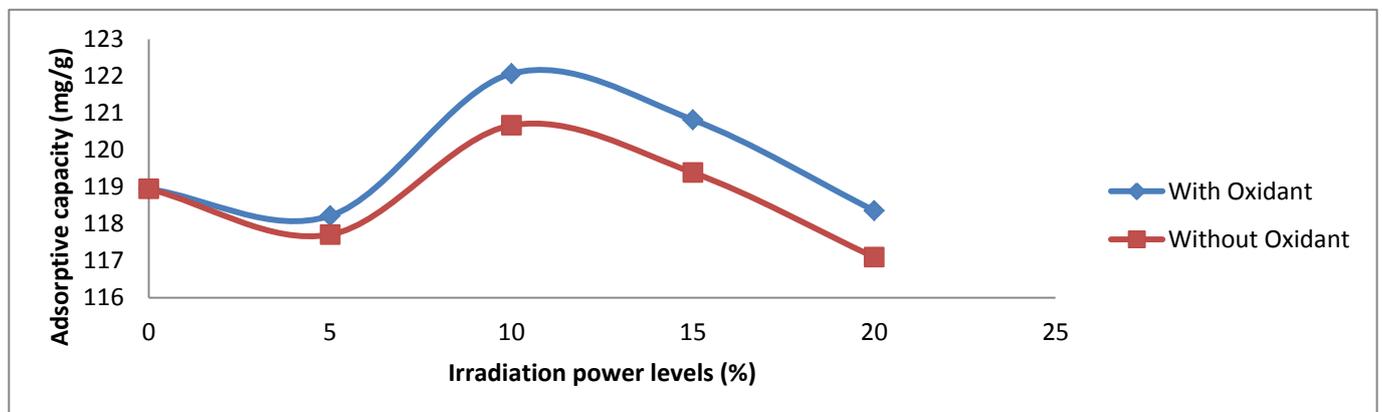


Figure 2. Effect of Microwave radiation time on the adsorptive capacity of regenerated activated carbon

After saturation, Swiss blue dye loaded activated carbons were exposed to microwave irradiation at

81% power level corresponding to 719W for 10 minutes. The effectiveness of the radiation on the activated carbons was measured by adsorbing the

same concentration of Swiss blue dye. The impact of six cycle of adsorption -regeneration was studied and the adsorptive capacity and regeneration efficiencies calculated. The regeneration was studied with 30% H<sub>2</sub>O<sub>2</sub> as an oxidant. Figure 3 shows the effect of cycle numbers on the adsorptive capacities of the regenerated adsorbents. It was observed from the plots, that the adsorptive capacities of the regenerated activated carbons increased with increase in initial dye concentration for all the cycles. This shows the effectiveness of microwave heating in desorbing the adsorbates. The decrease on the adsorptive capacities as the number of cycles was increased can be attributed to the weakening of the pores as a result of successive heating.

The effect of number of cycles on the regeneration efficiencies (figure 4) shows that the regeneration efficiencies decreased as number of cycles and initial dye concentrations were increased. It was equally observed that the regeneration efficiencies were slightly above hundred at 100mg/l initial dye concentration for cycles (1 – 4). For higher initial dye concentrations, it was observed that the adsorptive capacities were slightly preserved as observed on the regeneration efficiencies which were slightly less than 100.

This phenomenon might be attributed to the reorganization of internal structures as a

consequence of the adsorbate molecules evolving during the microwave irradiation stage [13]. This finding was in agreement with the work done by Dehdashti et al [27] on the regeneration of granular activated carbon saturated with gaseous toluene by microwave irradiation. Dehdashti [27] observed that after five repetitive treatment cycles by microwave energy, the adsorption capacities remained approximately constant at higher power levels, after which slight reduction was observed at subsequent cycles. . Preservation of adsorptive capacity by microwave heating implied, improvement in surface and porosity characteristic as a result of the radiation. The surface area, mesopore volume and mesopore volume of activated carbon regenerated with microwave for 15 minutes increased with increase in treatment cycles [28]. His findings confirmed previous literature on the effect of microwave irradiation on the textural properties of carbon [24]. It can be recalled that mesopores was responsible for adsorption of Swiss blue dye, thus increase in adsorptive capacity was recorded for microwave radiation on spent activated carbon.

The lower regeneration efficiencies recorded at higher initial dye concentration can be attributed to initial solid phase concentrations at higher initial dye concentrations which were not properly described.

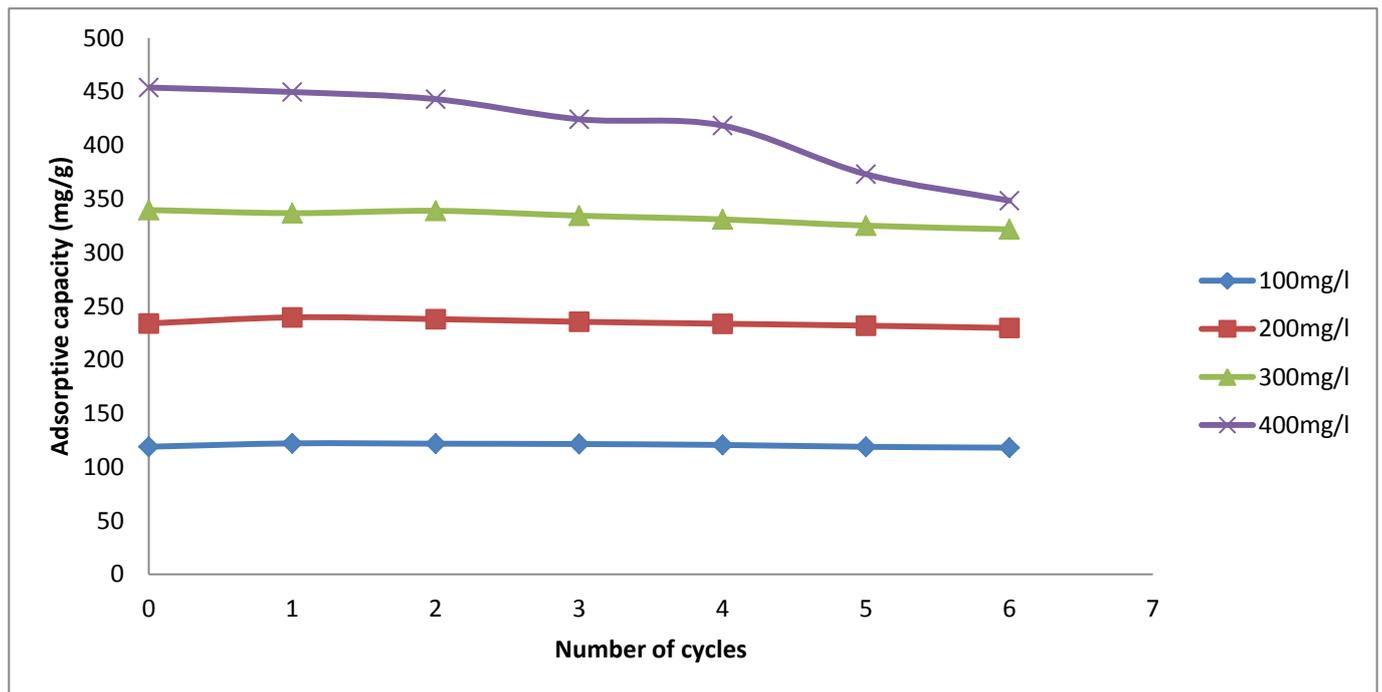


Figure 3. Effect of Microwave Regeneration cycles on the Adsorptive capacity of the spent activated carbons.

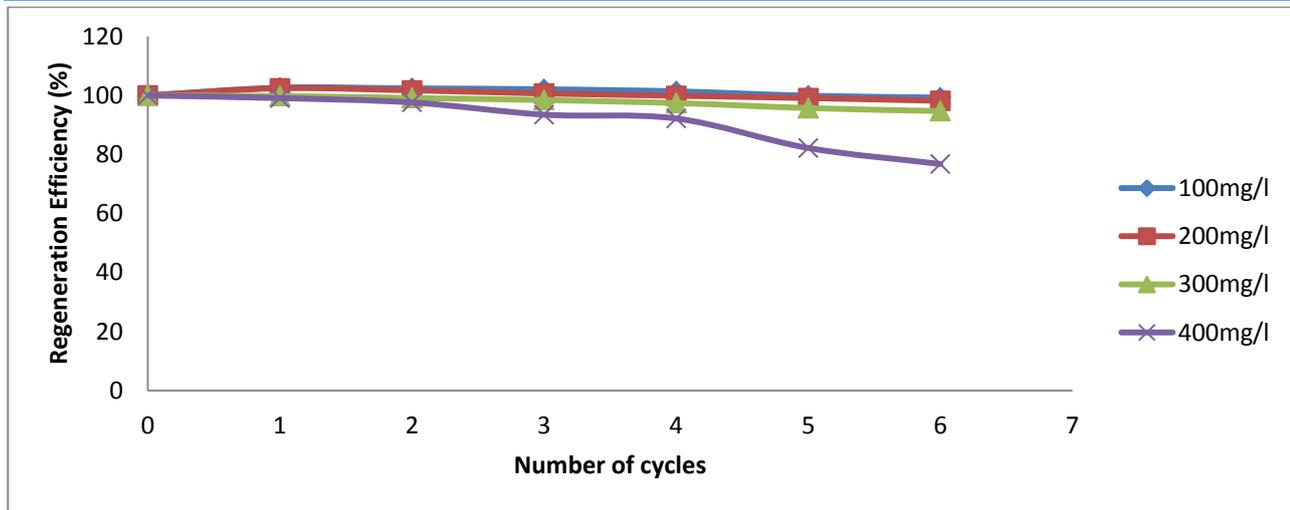


Figure 4. Effect of Microwave Regeneration cycles on the Regeneration efficiency of the spent activated carbons

#### e) Characterization of the activated carbon

The activated carbons used in this study were characterized before adsorption and after microwave regeneration. This was undergone to understand the effect of microwave regeneration on the properties of the regenerated carbon. The characterization was done after six cycles of regeneration using 100mg/l initial dye concentration. The results of the characterisation were showed on Table 1. It was observed that surface groups dominating the activated carbon prior to adsorption were acidic groups.

This increased the number of acid site on the activated carbon. The use of chemical reagents (KOH) in the activation process provided an increase in the amount of acid groups present in the activated carbon surface as observed in other studies [29], [30], [31]. The pH<sub>pzc</sub> obtained using pH drift method equally provided important information about the characteristics of acidity and basicity of activated carbon [31]. According to our results, pH<sub>pzc</sub> within 6.3 showed that acidic sites dominated the carbon. The pH<sub>pzc</sub> equally helped in understanding the mechanism of adsorption. pH of sample greater than pH<sub>pzc</sub> will adsorb on the carbon. This is why Swiss blue dye been a cationic dye adsorbs on the on the prepared activated carbon. This was in agreement with the work done by other researchers that produced activated carbons from chemical activation [30],[32], [23]. It was observed that after regeneration, the acidic characters changed to basic characters. According to Cazetta et al [31], basic characters dominate carbon produced from thermal treatment. That is why it was observed that the basic character of the microwave regenerations was high. Basic character of microwave regenerated samples was high because of the dispersive interaction between the adsorbed Swiss blue dye with the π-electron density of the carbon samples by means of microwave induced treatment [33]. Equally, oxidant used in the regeneration has power to remove

surface functional groups of activated carbon [34]. The regeneration decreased the oxygen surface functional groups with a subsequent increase in pH<sub>pzc</sub> values of the regenerated sample. This change in pH<sub>pzc</sub> can still favor the adsorption of cationic dyes [23]. Another changes observed was on the surface area and iodine number of the regenerated sample. The extensive network of pores throughout activated carbon particle provides the larger surface area for adsorption to occur. The high surface area after regeneration with microwave suggested that microwave exposure resulted in an increase in surface activation of the carbon. During microwave regeneration, new pores developed and existing pores were either widened or deepened. The result was an increase in surface area. The increase in surface area also indicated that excessive burn-off or surface oxidation was minimal during microwave regeneration [35]. This shows that after successive adsorption – regeneration cycles, the adsorptive capacity of microwave regenerated carbon was preserved [23], [27].

FTIR spectra of microwave regenerated activated carbon indicated the disappearance of strong bond around 1430cm<sup>-1</sup> after regeneration (Figure 5). The spectrum of regenerated samples had fewer functional groups compared to unused activated carbon. This is as a result of further elimination of surface functional groups and volatile matters as a result of the regeneration method used. It can equally be ascribed to the oxidant used for the regeneration which removed the surface function groups. This confirmed the earlier results obtained on the characterization of the regenerated samples.

#### IV. CONCLUSION

This study has demonstrated the effectiveness of conventional and microwave activation for production of activated carbon from hamburger seed shell and brewers' spent grain and the regeneration of spent activated carbons using chemical regeneration, microwave regeneration and conventional regeneration.

Table 1. Characterisation of Raw and Microwave Regenerated Activated Carbon.

Parameters	Units	Raw Activated carbon	Microwave Regenerated activated carbon
Surface Area	m <sup>2</sup> /g	518.3	510.2
Iodine Number	mgI <sub>2</sub> /g	1068.0	1006.1
Moisture Content	%	3.5	3.8
Ash	%	3.4	4.5
Volatile Matter	%	3.8	3.0
Fixed carbon	%	89.3	88.7
pH	-	6.5	6.8
pHzpc	-	6.2	8.6
Bulk Density	g/ml	0.49	0.47
Basic Sites	mmol/g	0.99	3.3
Acidic Sites	mmol/g	3.5	0.82

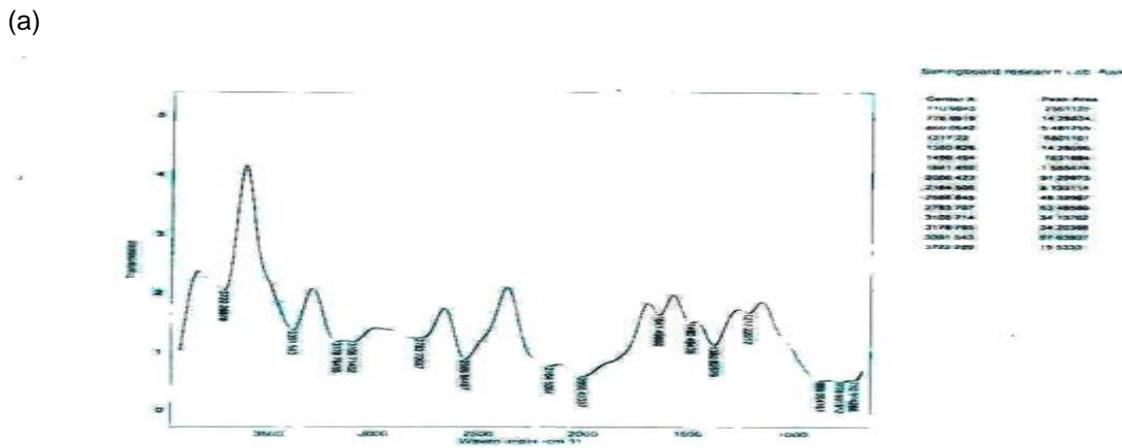
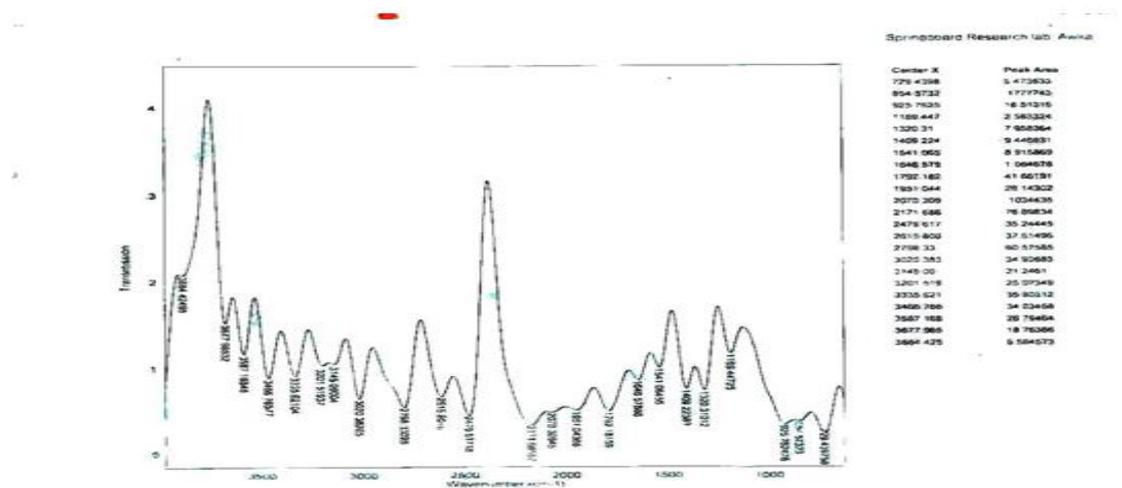


Figure 5. FTIR analysis of (a) raw activated carbon (b) Microwave regenerated activated carbon

The conclusions drawn from the results of this study are listed as follows:  
 Microwave can effectively be used to regenerate activated carbon from brewers' spent grain.  
 Microwave, regenerations studied using an oxidant (30% H<sub>2</sub>O<sub>2</sub>) showed that increase in regeneration cycle and initial solid phase concentration decreased the regeneration efficiencies and adsorption capacity

of regeneration carbon. Microwave regeneration relatively maintained the regeneration efficiency for lower initial solid phase concentration.

Characterization of the raw and regenerated activated carbon showed that the surface areas, iodine numbers, pH<sub>zpc</sub>, basic and acid sites of the carbon were affected as a result of the regeneration. These changes showed that the microwave

regeneration preserved the adsorptive capacity of the activated carbons.

## V. ACKNOWLEDGEMENT

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## REFERENCE

- [1]. Tharapong, V., Terachai, S., Somsak, D. (1999). Production of activated carbon from palm oil shell by pyrolysis and steam activation in a fixed bed reactor. *Science Asia*, Vol. 25, 211-222.
- [2]. Jones, D.A., Lelyueld, T.F., Manrofidies, S.D. (2002). Microwave heating application in environmental engineering a review. *Resource conservation and recycling*. 34 (2), 75-90.
- [3]. Savador, F., Sanchiz, J.C. (1996). A new method for regeneration of activated carbon by thermal desorption with liquid water under subcritical condition. *Carbon*, 34(4), 511-516.
- [4]. Chen, M.S., Wang, J.H.(2006). Microwave regeneration of activated carbon containing toluene. *Environmental pollution control technology and equipment* 7(6). 7 7-99.
- [5]. Zhang, H.P., Ye, L.Y. (2001). Electro-chemical regeneration of activated carbon. *Chem. Eng. Progress* 20 (1), 17-20.
- [6]. Mundale, V.D., Joglekar, H.S.(1991). Regeneration of spent activated carbon by wet air oxidation. *Chem. Eng.* 69, 1149-1159
- [7]. Menendez, J.A., Menendez, E.M., Iglesias, M.J., Garcia, A., Pis J.J. (1999). Modification of the surface Chemistry of active carbon by means of microwave induce treatment. *Carbon* 32, 115-1121.
- [8]. Hejazifar, M., Azizian, S., Sarikham, H., Li, Q., Zhao, D.Y. (2011). Microwave assisted preparation of efficient activated carbon from grape vine rhytitious for the removal of violet from aqueous solution. *J. Anal. Appl. Pyrolysis*, 92, 258-266.
- [9]. Ania, C.O., Menendez, J.A., Parra, J.B., Pis, J.J (2004). Microwave induced regeneration of activated carbon polluted with phenol. A comparison with conventional thermal heating regeneration. *Carbon* 42, 1377-1381.
- [10]. Brewers' Guardian (2011). *Brewers' Grains. Opportunities Abound Advantage Publishing Ltd*
- [11]. Hameed, B.H., Salman, J.M., Ahmad, A.L. (2010). Adsorption isotherm and kinetic modeling of 2,4-D pesticide on activated carbon derived from date stones. *Journal of Hazardous Materials*, 163(1), pp.121-126.
- [12]. Bradshaw, S. M., Van Nyk, E.J., DE Swardt, J.B (1998). Microwave heating principles and the application to the regeneration of granular activated carbon. *The journal of the South African Institute of Mining and Metallurgy*, July/ August 201.
- [13]. Foo K.Y., Hameed B.H. (2012). Microwave-assisted regeneration of activated carbon. *Bioresour. Technol.* 119, 234-240.
- [14]. Ekpete, O.A., Horsfall M. J. N. R. (2011) Preparation and characterization of Activated carbon derived from fluted pumpkin stem waste (*Telfairia occidentalis* Hook f) *Res.J.Chem Sci.* Vol 1(3)
- [15]. Rengaraj ,S., Seung -Hyeon, M., Sivabalm, S.(2012). Agricultural solid waste for the removal of organic adsorption of phenol from waste and waste water by palm seed coat activated carbon. *Waste management* 22. 543-548
- [16]. Gimba C., Musa. I. (2007). Preparation of activated carbon from agricultural waste . cyanide binding with activated carbon matrix from coconut shell. *J. Chem. Nigeria* .32. 167-170.
- [17]. Onwu D. O. (2000). *Coal fundamental and conversion technology*. Immaculate publications Enugu, Nigeria.
- [18]. Azubuike C. P., Rodriguez H., Okhamafe A. O., Rogers R.D. (2012) Physicochemical properties of maize cob cellulose powder reconstituted from ionic liquid solution, *cellulose* 19, 425-433.
- [19]. Sears , G.W.(1976). Determination of specific surface area of colloidal silica by titration with sodium hydroxide. *Analytical chemistry*, 28:1981-1983
- [20]. Sun kang, Jiang Jian-chun. Jun-ming Xu. (2009). Chemical regeneration of Exhausted Granular activated carbon used in citric acid fermented solution Decoloration. *Iran chem. chem. Eng.* Vol.28, No.9
- [21]. Ania, C.O., Parra, J.B., Menendez, J.A.(2005). Effect of microwave and convectional Regeneration on the microporous and Mesoporous Network and on the Adsorptive capacity of Activated carbons. *Micropor. Mesopor. Mater.*, 87, 7-15.
- [22]. Guocheng, L.V., Lime, WU., XiaoyuWang, Libing LIGO, Zhauhui LI., Wenhui He (2012).

Regeneration of Carmel Saturated Activated carbon Jointly by Microwave and Extractive Method International Journal of Chemical Reactor Engineering.

[23]. El-Sayed, Y.M., Bandoz, J.J. (2004). Adsorption of Valeric Acid from Aqueous Solution Unto Activated carbon: Role of Surface Basic Sites, J. Colloid Interface Sc. 273, pp. 64 -72.

[24]. Vargas, A. M.M., Cazetta, A.L., Garga, C.A., Moraes, J.C.G., Nogami, E.M., Lenzi, E., Costa, W.F., Almeida, V.C. (2011). Preparation and characterization of Activated carbon from a New Raw Lignocellulosic material: Flamboyant (*Delonix regia*) Pods. J. Environ, manage, 92; pp. 178 – 184.

[25]. Cazetta, Andre, L., Alexandro, Vargas, M., Eurca, Nogami,M., Marcos Kunits, H., Marcos Guilherme, R., Alessandro Martina, C., Tais Silva, L., Juliana Moraser, C.G., Vitor Almerda, C. (2011). NaOH – Activated carbon of High Surface Area Produced from Coconut Shell: Kinetics and Equilibrium Studies from the methylene Blue Adsorption Chemical Engineering Journal. 174, pp. 117 – 125.

[26]. Hameed, B.H., Mahound, B.K., Ahmad, A.L. (2008).Equilibrium Modeling and kinetic Studies on the Adsorption of Basic Dye bya Low-Coast Adsorbent: Coconut (*Cocos nulifera*) Bunch Waster, J. Hazard, Mater, 158, pp. 65 – 72.

[27]. Radovic, L.R., Silva, I.F., Ume, J.I., Menendozi, J.A., Leon, C.A., Leon, Y.A., Scaroni, A.W. (1997). An Experimental and Theoretical Study of the Adsorption of Aromatics Possessing Electron - Withdrawing and Electron – Donating Carbon 35, 1339- 1349.

[28]. Sun kang, Jiang Jian-chun. Jun-ming Xu. (2009). Chemical regeneration of Exhausted Granular activated carbon used in citric acid fermented solution Decoloration. Iran chem. chem. Eng. Vol.28, No.9

[29]. Surfnarski Micheal David, B.S (1999). The regeneration of granular activated carbon using Hydrothermal Technology. Thesis presented to the faculty of the graduate school of the University of Texas at Austin.