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## Reactivation and recycling of spent carbon using solvent desorption followed by thermal treatment (TR)

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**Abstract** This study demonstrated a technique to regenerate spent activated carbon using solvent desorption followed by thermal decomposition of pollutants. Dichloromethane is used as solvent for desorption and thermal reactor under inert ( $N_2$ ,  $CO_2$ ) atmosphere used for thermal reactivation of the spent carbon. Physical, chemical, functional chemistry and thermal behavior of the samples before and after treatment are featured by means of pH, bulk density, moisture content, ash content, Fourier transform infrared spectroscopy, thermo-gravimetric differential thermal analysis. The adsorptive property of the activated spent carbon is quantified using methylene blue and iodine as model compounds. After reactivation, methylene blue and iodine number adsorption is improved from 5 to 96 % and from 10 to 99 %, respectively. This regenerated carbon applied for paper mill and pharmaceutical effluents. 95 and 94 % of the COD reduction and color removal are observed by spent reactivated carbon.

**Keywords** Spent carbon · Solvent · Desorption · Thermal reactor · Reactivation

### Abbreviation

ASTM	American Society for Testing and Materials
AWWA	American Water Works Association
CEFIC	European Council of Chemical Manufacturer Federations
APHA	American Public Health Association

### Introduction

Environmental pollution is the major problem associated with rapid industrialization, urbanization and rise in living standards of people. Waste seems to be a by-product of growth with industrialization. Hazardous waste is the severely concerned issue which causes not only environment pollution, but also several ill health effects on human beings. A country like India can ill-afford to lose them as sheer waste. On the other hand, with increasing demand for raw materials for industrial production, the non-renewable resources are dwindling day by day. Therefore, efforts are to be made for controlling pollution arising out of the disposal of wastes by conversion of these unwanted wastes into utilizable raw materials for beneficial use. The problems relating to disposal of industrial solid waste are associated with lack of infra-structural facilities and negligence of industries to take proper safeguards [3, 8, 31].

The exhausted activated carbon is called “spent carbon”. Due to pollutants load it comes under hazardous waste category. Generally spent carbon is disposed off in regular landfills which results in hazardous materials seeping into the ground, thus polluting underground and surface water sources. Many landfills hence require counter-measures

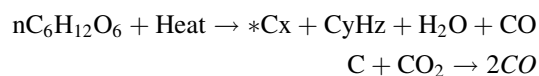
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against groundwater contamination, an example being installing a barrier along the foundation of the landfill to contain the hazardous substances that may remain in the disposed waste [7, 37]. As spent carbon is a type of flammable waste, it can sometimes be burned as energy source. It has been “destroyed” by incinerating it at a high temperature. However, this type of treatment releases toxic gases produced by the combustion of by-product or other materials. The problem of solid waste incinerator is the emission of dioxins as well as the bad smell with combustion [20]. In addition, the use of activated carbon is economically limited, since the cost of carbon for single use is very high. The process of carbon recycling reduces operating costs since the cost of reactivated carbon is typically 20–40 % less than the original cost of activated carbon. Also, it eliminates the liability associated with handling and disposal of spent carbon [11, 13].

The reactivation or the regeneration of activated carbon involves restoring the adsorptive capacity of saturated activated carbon by desorbing adsorbed contaminants on the activated carbon surface. Generally, solvent regeneration processes are applicable to methods with reversible adsorption (desorption), such as high organic concentration wastewater treatment and recycled wastewater containing precious metals [10]. Solvent desorption of pollutants has been used for many years as a method of concentrating for subsequent process, with chloroform, and ethanol being recommended by American Public Health Association (APHA) [9, 18]. In any solvent regeneration scheme, once the solvent has been used to desorb the primary adsorbate from the carbon, the effective recovery of the solvent in reasonably pure form by distillation or flushed water bed method. In this study, dichloromethane is selected as a solvent for desorption treatment [10, 17]. Followed by this, thermal method has been applied for reactivating the spent carbon.

Thermal reactivation is the most suitable spent activated carbon regeneration technique for industrial processed activated carbon (hazardous character) [6]. In the thermal regeneration process, the spent carbon [11] has been dried at approximately 105, 500 °C under an inert atmosphere applied for temperature desorption, and finally residual organic gasification by an oxidizing gas (carbon dioxide) at elevated temperature 800 °C has been applied [28].



where \*C<sub>x</sub>, charC<sub>y</sub>H<sub>z</sub>, volatile hydrocarbons

%Regeneration of the carbon

$$= \frac{\text{Adsorption capacity after regeneration}}{\text{Adsorption capacity of fresh carbon}} \times 100.$$

**Table 1** Analytical methods

S. no.	Parameter	Method
Analytical parameters		
1	Bulk density	Standard method no: ASTM D 2854
2	pH	Standard method no: ASTM D 3838
3	Moisture content	Standard method no: ASTM D 2867
4	Ash content	Standard method no: ASTM D 2866
5	COD	Standard method no: APHA 5220C
Adsorption tests		
1	Methylene blue	Standard method no: CEFIC ACM 54
2	Iodine number	Standard method no: AWWA B 600

## Materials and methods

### Reagents

Dichloromethane (DCM), iodine, methylene blue and KBr are all procured from S.D.F.C.L, India. Nitrogen and carbon dioxide gases were supplied by BOCIL, India.

### Analytical methods

Physicochemical parameters (Table 1) such as pH, bulk density, ash content and moisture content were determined in accordance to standard methods (APHA 1998).

### Spent carbon

The hazardous spent activated carbon sample was collected in airtight bags from the pharmaceuticals industry at Jeedimetla industrial park, Hyderabad, Andhra Pradesh, India. After collection the sample was air dried for 2–3 h in order to dewater the sample.

### Methodology

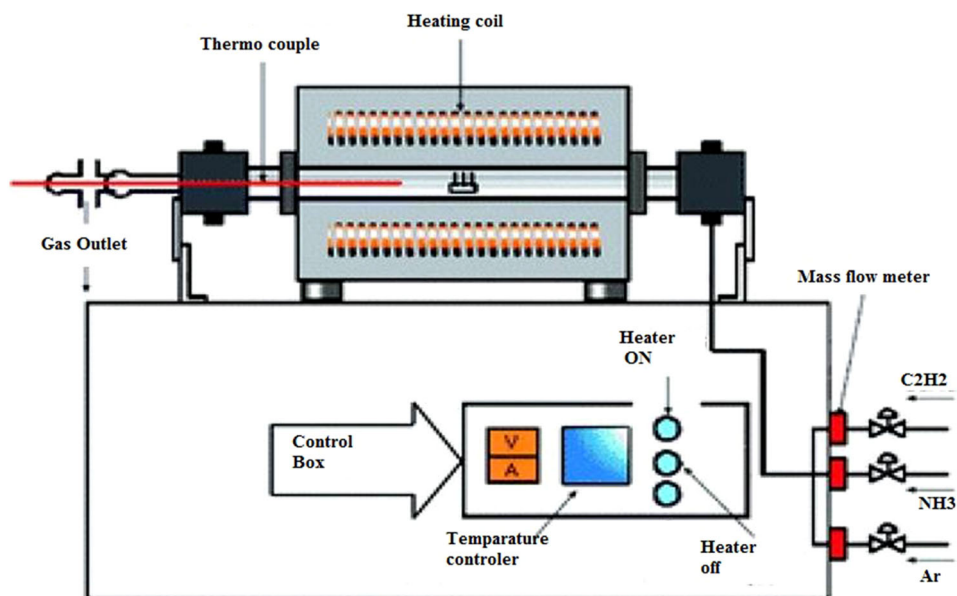
#### *Solvent desorption*

Five grams of the air-dried sample was dispersed in 100 ml of solvent (DCM) and stirred for 30 min. The solution was allowed to settle for 20 min after which, it was filtered using Whatman filter paper no. 44. The retained carbon on the filter paper was collected and kept in hot air oven at 105 °C till the sample reached a constant weight. While this dried carbon was thermally regenerated in a TR unit, the spent solvent was recovered by distillation method.

#### *Thermal reactivation of spent carbon*

Thermal reactivation was carried out in thermal reactor unit (TR), the design of which is given in Fig. 1. TR consists of a

Fig. 1 TR-reactor



100-cm-long quartz tube with inner diameter of 1.2 cm and a boat of 10 cm length into which the sample was loaded. The reactor is provided with an inlet pipe for the flow of gases and an outlet pipe for removing of toxic gases produced during reaction. An electric oven is used to heat the reactor to desired temperatures. Approximately 5 g of spent carbon was filled into the boat and kept in a reactor zone of TR. Air spaced quartz tube was purged with the flow of nitrogen ( $N_2$ ) gas at a flow rate of 500 ml/min till the reactor temperature rose to 500 °C. After the temperature reached 500 °C,  $N_2$  gas was replaced with carbon dioxide ( $CO_2$ ) and continued until the temperature reached 900 °C. At 900 °C reactivation was carried for 30 min. The electric oven was then switched off and allowed to cool. This reactivated carbon is stored in airtight bottle for further examinations.

#### *Application of reactivated spent carbon in the treatment of industrial effluents*

To find the efficacy of the reactivated spent carbon (RAC), adsorption experiments were carried out for the removal of color in paper mill effluent and COD removal in pharmaceutical effluent.

In these experiments, 1 g of activated carbon was taken in stoppered bottles containing 100 ml of untreated wastewater at 25 °C and then kept for shaking. Samples were collected and analyzed for color and COD removal. Finally, a comparison was made among all the activated carbons to evaluate their performance efficiency.

#### Characterization of the samples

Samples were characterized for color removal using double beam Shimadzu UV 2450 UV–visible Spectrophotometer at

a wave length of 600 nm. The presence of functional groups in all types of activated carbons was determined using Perkin Elmer Fourier transform infrared spectroscopy (FTIR). Wave length number ranged from 4000 to 400  $cm^{-1}$ . The thermal behavior and weight loss of the sample were determined using thermo-gravimetric differential thermal analysis (TG-DTA). TG-DTA of the sample was done using S-II EXSTAR-6000, TG/DTA-6300 thermal analyzer. Sample was loaded in aluminum container and heated to 800 °C at the rate of 2 K/min with air circulating at a speed of 150 ml/min. Elemental compositions analysis (energy dispersive X-ray analysis, EDAX) was carried with HITACHI S 3400N scanning electron microscope. The composition of carbon sample information was produced from signals of electrons that interacted with the atoms present in the sample.

## Results and discussion

The results of the investigation are presented below and discussed in detail.

#### Characterization of activated carbon

Characteristics like bulk density, pH, moisture content and ash content of the samples of commercial activated carbon (CAC), spent activated carbon (SPAC), DCM desorbed activated carbon (DCMAC) and thermal reactivated carbon (RAC) are shown in Table 2. The bulk density of powdered activated carbon relates to the physical weight per volume of powder and was generally measured in milligrams/cc. Higher volume of carbon can deliver a higher surface area to the water for adsorption of micro-pollutants [2, 24, 29]. The bulk density of the CAC, SPAC, DCMAC and RAC

**Table 2** Physico-chemical analysis of commercial, spent, DCM, RAC

S. no.	Name of the experiment	CAC	SPAC	DCM AC	RAC
1	Bulk density (mg/cc)	0.40	0.59	0.55	0.44
2	pH	6.7	7.95	6.4	6.9
3	Moisture content (%)	7	24	15	7
4	Ash content (%)	2.6	6	6	4

was observed to be 0.40, 0.59, 0.55 and 0.44, respectively, with only slight variance between CAC and RAC (0.04).

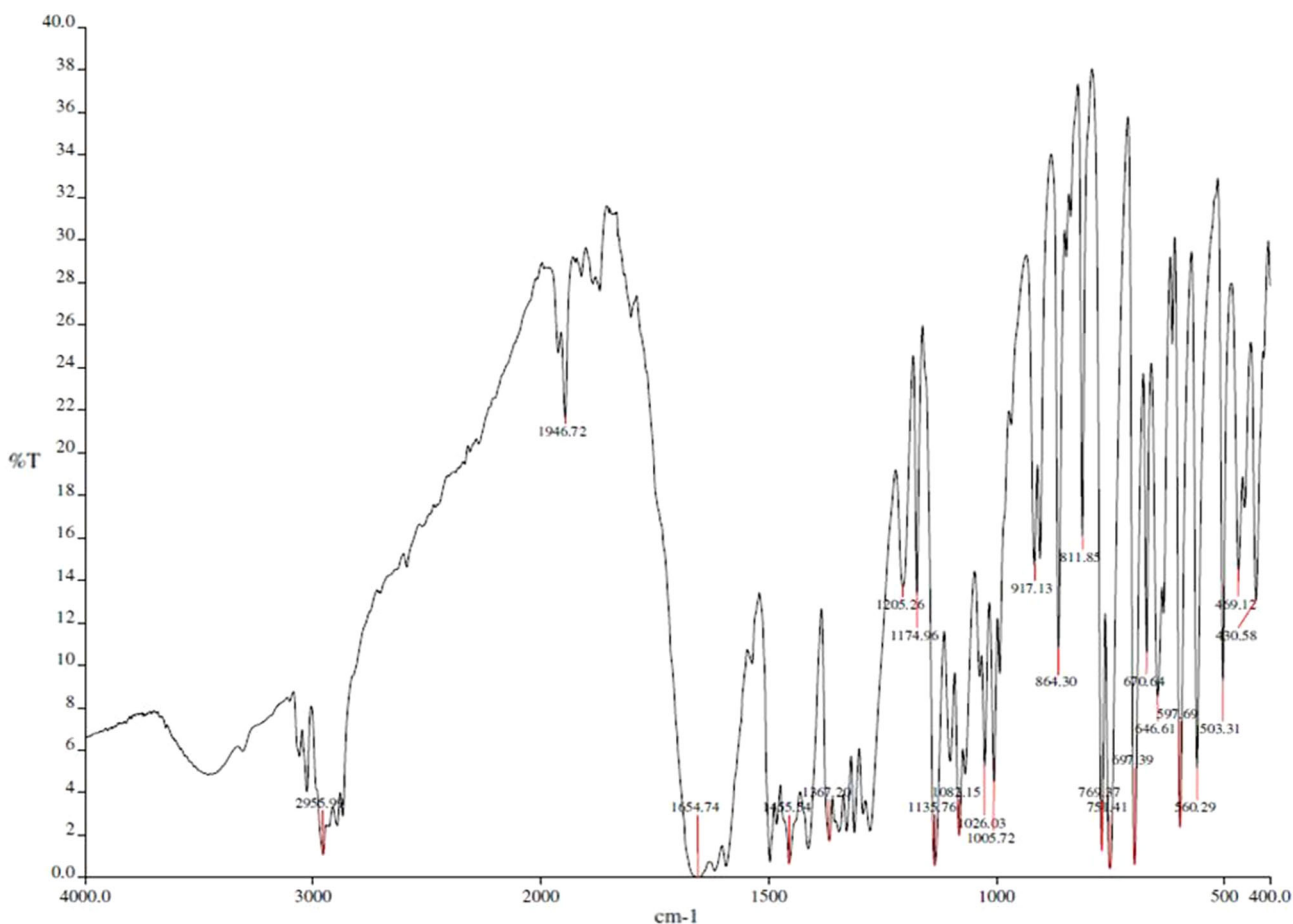
The pH of RAC and CAC was found to be 6.9 and 6.7, respectively. pH from 6 to 8 is acceptable for different applications as adsorbent according to Ahmedna and Okieimen [1, 26].

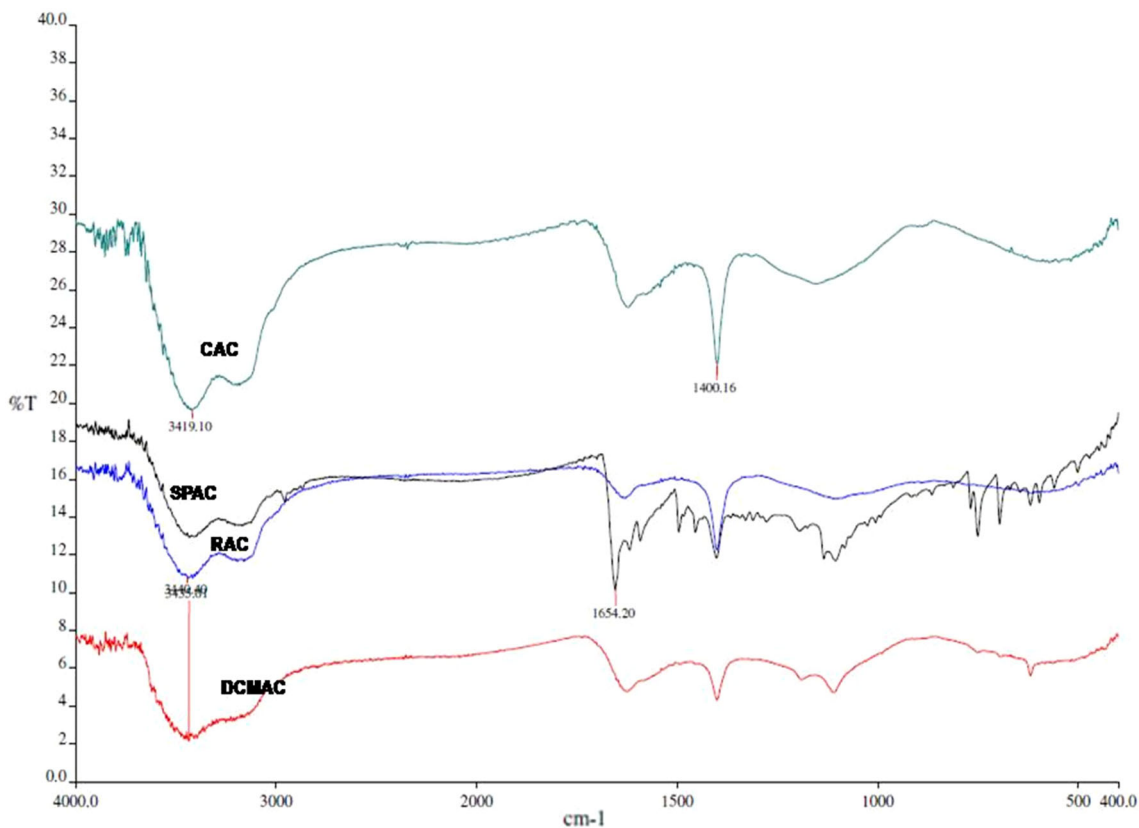
The moisture content (MC) of activated carbon is often required to define and express its properties in relation to the net weight of the carbon. High moisture content is undesirable, since it has low adsorptive capacity [34]. Moreover, it also adds transport and storage costs. SPAC has 24 % MC, after reactivation (RAC) it reduced to 7 %, which is equal to MC of CAC. DCMAC had a MC of 15 %.

The ash content influences the ignition point of the carbon and may be a major consideration where adsorption of certain solvents is concerned since the lower the ash value, better the activated carbon for use as adsorbent [35]. In the present study, the ash results show that reactivation reduces the ash levels of carbon. The reduction in ash could be attributed to volatilization of constituents at higher temperature that could lead to a reduction in ash content [10, 16]. In the present study, the total ash content of 6 % is observed in DCMAC and SPAC samples while the CAC contained 2.5 % ash content (Table 2) and RAC is observed to contain 4 % of the ash content. The high percentage of ash content in RAC compared to CAC might be due to the partial burning of the inorganic and organic compounds in RAC while in CAC they are initially adsorbed on to the activated carbon.

#### Adsorption tests

This is the most fundamental parameter used to characterize activated carbon performance. It is a measure of

**Fig. 2** FTIR analysis of the DCM residue



**Fig. 3** FTIR analysis of the carbon samples

activity level and it predicts the suitability for a certain treatment objectives.

#### *Methylene blue adsorption*

Methylene blue identifies the capacity of an activated carbon to decolorize the aromatic dye compounds having medium molecular size (2–5 nm). It is a quick method for comparing different batches of activated carbon of the same quality. Methylene blue has strong adsorption onto solids and its recognized usefulness in characterizing adsorptive material [21]. The methylene blue adsorption of CAC, SPAC, DCMAC and RAC were observed to be 280, 15, 115 and 270 mg/g.

#### *Iodine number*

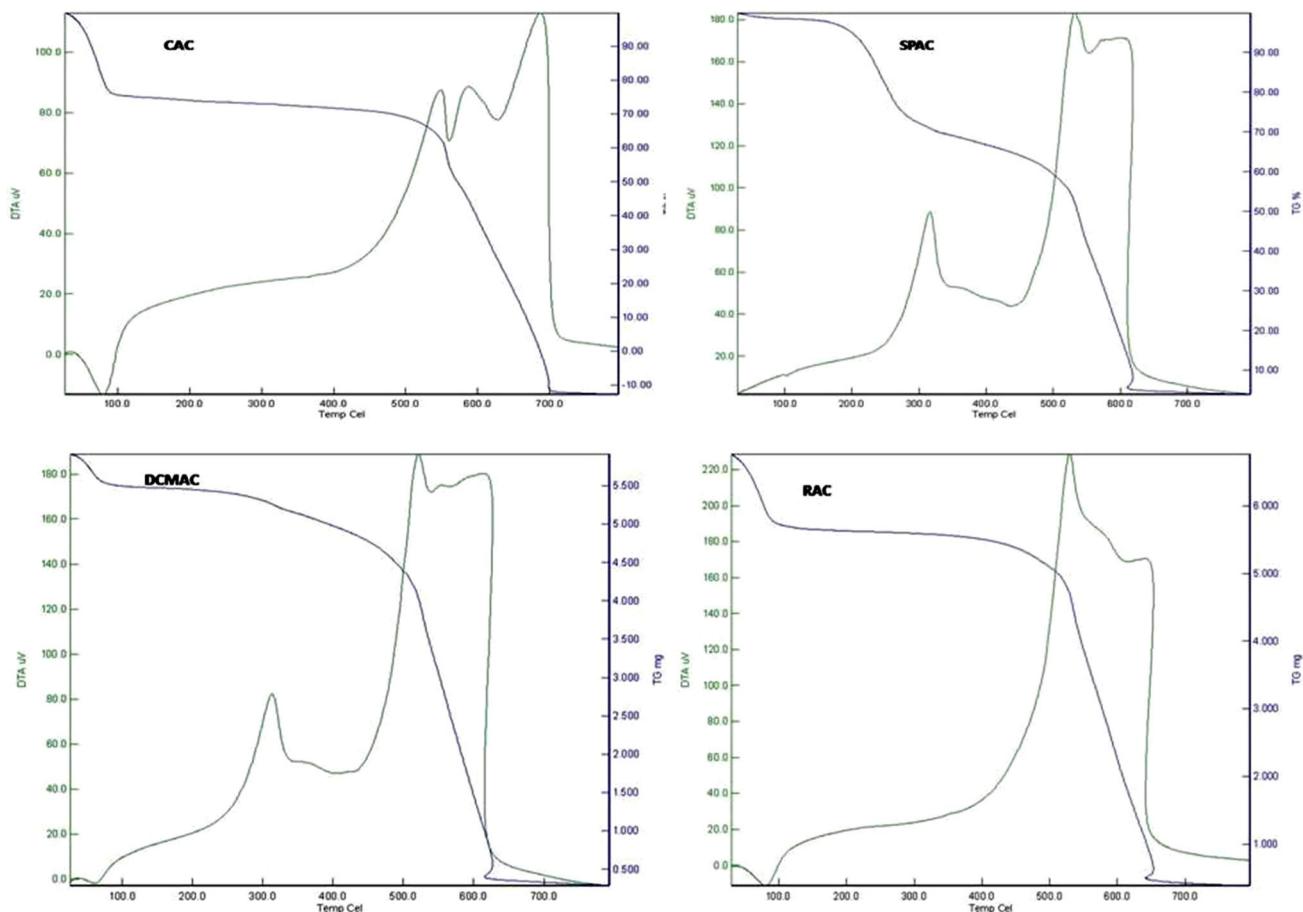
Iodine is a small molecule which will be well adsorbed on to the activated carbon. It is a measure of the microspore content of the activated carbon (values >0–20 Å, or up to 2 nm) by adsorption of iodine from solution. Sorbents with high iodine number performs better in removing small-sized contaminants [5, 23]. The iodine number of the RAC (1066 mg/g) is observed to be approximately equal to that

of CAC (1076 mg/g), which is a good indication for the reactivation of SPAC [15, 21].

#### Determination of functional groups of different carbons by FTIR

The FTIR analysis depicts the transmittance spectra of the CAC, SPAC, DCMAC, and RAC. SPAC spectra (Fig. 2) vibrations imply the presence of monomeric alcohols, phenols and also alkane compounds due to the medium and strong stretching vibrations which was seen at wave numbers corresponding to 3455, 3027, and 2955  $\text{cm}^{-1}$ , respectively [19, 36]. Strong stretching vibrations at 1654  $\text{cm}^{-1}$  might correspond to the C–H bonds of the phenyl ring substitution overtones. The presence of N–H bonds of the amines and O–H bonds of the carboxylic acids were also suspected. Strong and medium rocking vibrations at 751  $\text{cm}^{-1}$  might correspond to the presence of N–H groups in it [15, 25].

However, it is observed that the spectra of the SPAC and RAC vary by large deviations. But the spectra of RAC and CAC were observed to be almost similar, and might have few variations due to the presence of different functional groups on its surface. RAC and CAC (Fig. 3) spectra's



**Fig. 4** TG-DTA analysis of the carbon samples

vibration implies the presence of C–O and C–OH groups due to the strong stretching vibrations which correspond to the wave numbers 3419, 3440, and 1400  $\text{cm}^{-1}$ , respectively.

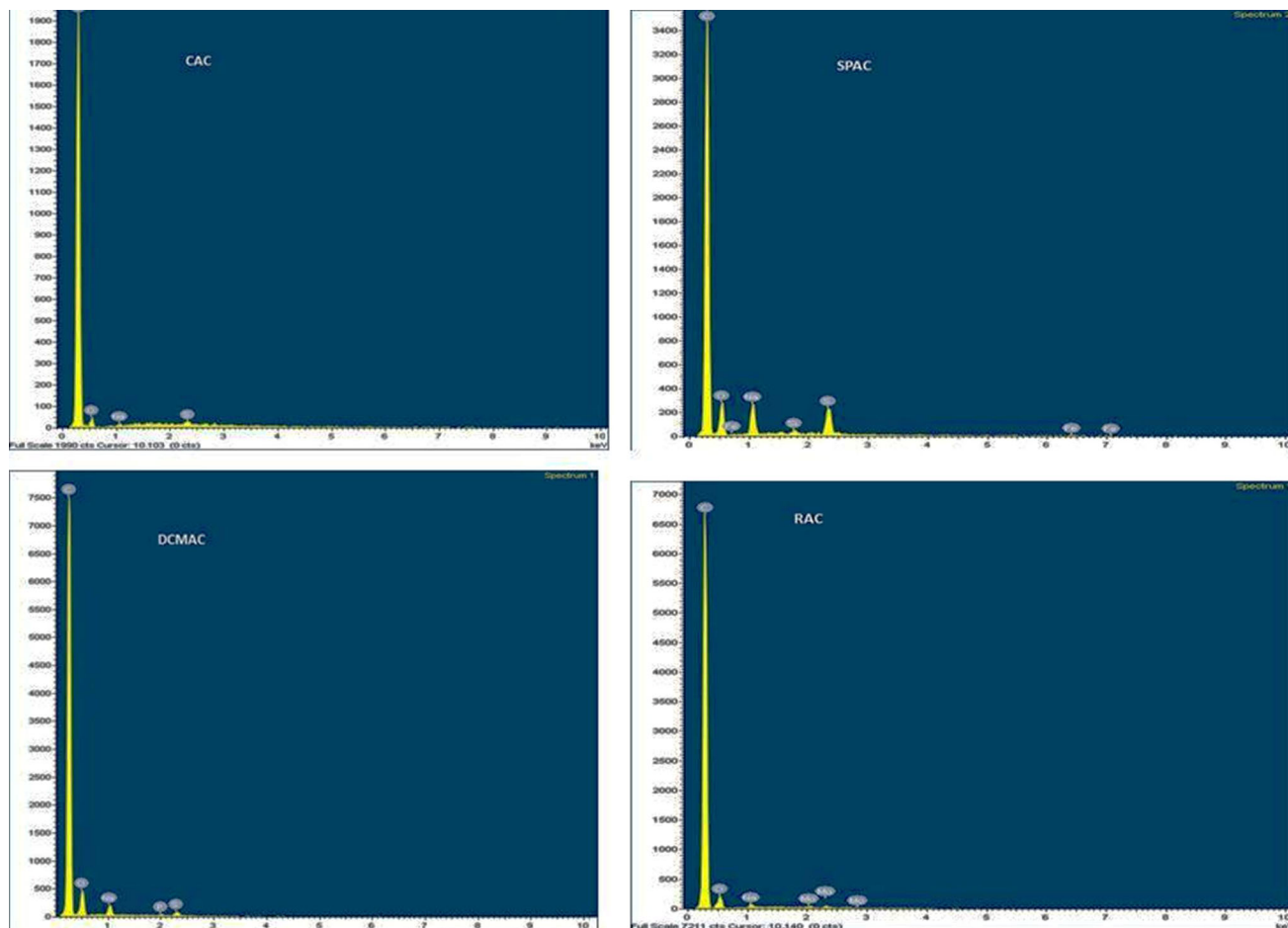
#### Thermo-gravimetric-differential thermal analysis (TG-DTA)

The horizontal axis represents the sample temperature and the vertical axis shows (Fig. 4) the TG (weight change) and DTA (thermal behavior). The bottom of the TG curve indicates a weight decrease and the top indicates weight increase. For the DTA curve, the downward direction indicates an endothermic reaction and the upward direction indicates an exothermic reaction [24, 30].

Three different zones were observed in the oxidizing atmosphere for all the carbons as shown in Fig. 4. For CAC, the first zone ranged from room temperature to 90 °C, the second zone from 90 to 550 °C, and the third zone after 550 °C. The maximum weight loss of 40 % was recorded in the second zone, while the first zone corresponds to comparatively low weight loss of 22 %. The

third zone showed almost complete weight loss. For SPAC only 6.1 % weight loss was observed in the first zone, from room temperature to 200 °C, 27 % weight loss in the second zone from 200 to 270 °C, and a weight loss of 46.6 % in the third zone from 270 to 510 °C. For DCMAC, the first zone ranged from room temperature to 90 °C, the second zone from 90 to 540 °C, and the third zone after 540 °C. The maximum weight loss of 27 % was recorded in the second zone, while the first zone corresponded to comparatively low weight loss of 8 %. The third zone showed almost complete weight loss. For RAC only 15.7 % weight loss was observed in the first zone, from room temperature to 90 °C, 29 % weight loss in the second zone, from 90 to 510 °C, and the third zone, after 510 °C a complete weight loss was observed. The first zone corresponded to removal of moisture and light volatiles. The weight loss was reported to be associated in part with the evolution of  $\text{H}_2\text{O}$ ,  $\text{CO}_2$  and  $\text{CO}$  [4, 22, 33]. Second zone is mainly attributed to the carbonization process of compounds and suggests the cracking reaction of C–C bonds and the third zone weight loss was due to ignition of the sample.





**Fig. 5** EDAX images of the carbon samples

In CAC three endothermic peaks appeared at 75, 580 and 650 °C, while only one exothermic peak appeared at 700 °C. For RAC one endothermic peak at 75 °C and two exothermic peaks have been observed at 520 and 640 °C. For SPAC exothermic peaks observed at 320, 540, and 610 °C. DCMAC endothermic peaks appeared at 50 and 535 °C. Exothermic reactions observed at 320, 520, and 610 °C. In observing the Fig. 4, the sharp exothermic peaks had a complicated organic structure [3, 25, 32].

#### Energy dispersive X-ray analysis

In order to obtain more information on reactivation of spent carbon, it was further analyzed using EDAX. Results are presented in Fig. 5 while the EDAX analysis revealed carbon activation in the reactivated sample (RAC). Practically the elements like C, O and Na were contained in all the samples. Carbon weight % content of the carbon samples (Table 3) were as follows: CAC (90.62 %) >RAC (88.72 %) >DCMAC (83.96 %) >SPAC (78.37 %). The

increase in the carbon weight % of the RAC compared to SPAC might be due to the volatilization of the pollutants during the reactivation process. This carbon increase in RAC is the evidence for reactivation of the spent carbon [12].

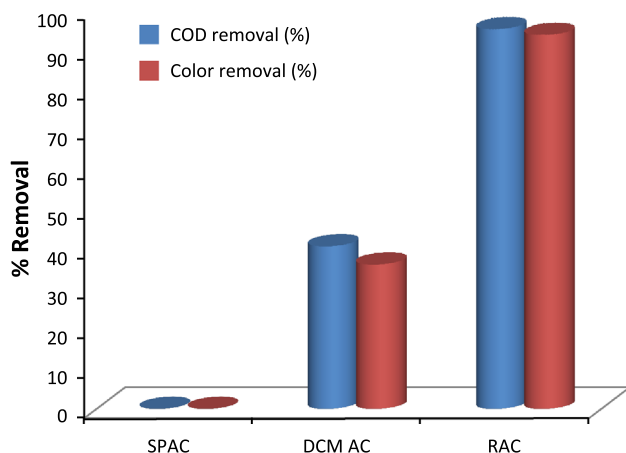
Applications of reactivated carbon in the waste water treatment process

Adsorption efficiency of the carbon was validated by applying for the treatment of pharmaceutical waste (organic content) and paper effluent (color contents). The efficiency was evaluated in terms of COD reduction for pharmaceutical waste water and color removal for paper effluent.

The efficiency of SPAC in the removal of color and COD was observed to be only 2 and 3 %, respectively. With partially reactivated DCMAC, the removal efficiency was observed to be 41 and 36 % for COD and color. A maximum removal efficiency of 95 and 94 % of the COD and color was achieved with RAC (Fig. 6). The

**Table 3** EDAX elemental representation of the carbon samples

S. no.	Name of the sample	Element	Weight %	Atomic %
1	CAC	C K	90.62	93.12
2		O K	8.28	6.39
3		Na K	0.46	0.24
4		S K	0.64	0.25
5	SPAC	C K	78.37	84.86
6		O K	14.68	11.94
7		Na K	2.97	1.68
8		Si K	0.28	0.13
9	DCMAC	S K	3.07	1.24
10		Fe K	0.63	0.15
11		C K	83.96	88.38
12		O K	12.7	10.04
13	RAC	Na K	1.65	0.91
14		P K	0.34	0.14
15		S K	1.35	0.53
16		C K	88.72	92.14
17	RAC	O K	9.52	7.42
18		Na K	0.5	0.27
19		Mo L	1.26	0.16

**Fig. 6** COD removal (%) and color removal (%) by SPAC, DCMAC and RAC samples

results thus indicate the increased adsorption capacity of RAC [14, 27].

## Conclusions

The study indicates that the regenerated activated carbon (RAC) by thermal regeneration could be a feasible and effective way of using the spent activated carbon. The adsorption capacity of RAC and CAC was comparable. This was proved by the iodine adsorption and methylene

blue number of RAC which were approximately equal to CAC. The FTIR spectral analysis also revealed the removal of functional groups after regeneration. RAC also showed good efficiency in the removal of color (%) and COD (%) of paper and pharmaceutical effluents. Since this is a lab-scale study, the study if validated for 2–3 cycles, can be extended to pilot scale.

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