

SURFACE AREA CHARACTERIZATION OF CARBON BLACK OBTAINED FROM WASTE TYRES

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ABSTRACT

Surface characterization of carbon black was done for untreated carbon black obtained from waste tyres using Pyrolysis. It chemically treated with Hydrochloric acid of different molar concentration (1M, 5M and 10M respectively). Acid demineralization process was carried out to decrease the inorganic impurities as well as to remove undesirable (ash) contents from the carbon black. The samples were demineralised for a period of 24 h. After 24 h the samples were filtered and thoroughly rinsed with distilled water to remove the residual acid. The samples were then dried. We also did BET of a KOH treated sample which was activated at 900 oC.

Keywords: Carbon Black; Pyrolysis; Molar Concentration; Demineralization; BET.

INTRODUCTION

The automobile has become an indispensable means of transportation for many households throughout the world. Thus, the disposal of vehicle tyres represents a major environmental issue throughout the world. Globally more than 330 million tyres are discarded every year and accumulated over the years in different countries (Cunliffe and Williams, 1999). In India, an estimated 350,000 tonnes of waste tyres per year are dumped mostly in an illegal process. The disposal and reprocessing tyres are difficult since they contain complex mixture of different materials such as rubber, carbon black, steel cord and other organic and inorganic minor components¹.

Fire hazards and large stockpiles could consequently cause uncontrollable burning and air pollution where it will be emitting large amounts of thick black smoke and noxious gases including carcinogens. Other than that, the current “conservation of natural resources concept”, the reuse (retreat) first, then reuse the rubber prior disposal, does not accommodate the ever increased dumping of tyres. In fact, due to the high cost of legal disposal for tyres, illegal dumping may increase. The disposal of tyres is also becoming more expensive, while this trend is likely to continue as landfill space becomes scarcer.

All of which have the potential to be processed into valuable products. The resulting hydrocarbons from thermal treatment can be used

directly as fuel. Added to petroleum refinery feedstocks. As for solid residue, the char can be used either as low-grade reinforcing² filler or as high carbon content material³⁻⁷. However, reprocessing of the char from pyrolysis of waste tyres into carbon black⁸ has been considered to be a profitable way to convert this material into valuable product.

Carbon black is one of the oldest manufactured materials, early uses can be traced back to ancient China, the early Egyptians and the production of Indian Inks. Early demand for carbon black was driven by the invention of movable type used in fifteenth century printing. The discovery that carbon black reinforces natural rubber and thereby greatly increasing the longevity of tires in the early nineteenth century thrust the material into the modern age. Today carbon black is found in all aspects of modern life. It is used in inkjet printer ink, as reinforcements for natural and synthetic rubber, it is the active agent in electrically conductive plastics and is used as a pigment and tinting-aid in paints, coatings, news paper inks and cosmetics to name a few.

EXPERIMENTAL PROCEDURE

Pyrolysis Procedure

Pyrolysis is the common name used for decomposing organic material at elevated temperatures in the absence of oxygen. The oxygen needs to be absent otherwise organic material may burn. Typically the process takes place under pressure and operating temperatures above 430 °C (800 °F). The word is originated from Greek based words “pyr” and “lysis” meaning “fire” and “separating”, respectively. Initial studies on pyrolysis of scrap tires have shown that tire-derived activated carbon, carbon black, boudouard carbon, and fuel gas are obtained.

Conversion Procedure

Carbon Black were produced from waste tyres through the conventional process of carbonization followed by steam or carbon dioxide activation. In the carbonization step, a cylindrical stainless steel atmospheric pressure reactor was used, provided with a ceramic furnace and power source heating system, gas-feed inlets, and accessories to collect the liquid and gas samples. A heat exchanger at the top kept the samples at room temperature until the start of each run when a suspension system lowered a basket containing the sample.

For each run, the basket was loaded with 20 g of scrap tyre and positioned in the cooling zone of the reactor, after a nitrogen flow was circulated through the entire system during 1 h to purge the air from the system. The heating system was switched on until the target temperature of 800 °C was reached. At this moment, the basket was lowered into the heating zone, then kept at this temperature during 1 hour. After this time, the heating system was switched off, and finally the sample was cooled at room temperature.

SAMPLE PREPERATION PROCEDURE

Samples were treated with Hydrochloric acid of different molar concentration (1M, 5M and 10M respectively) and KOH. Acid demineralization process was carried out to decrease the inorganic impurities as well as to remove undesirable (ash) contents from the carbon black. The samples were demineralized for a period of 24 h. After 24 h the samples were filtered and thoroughly rinsed with distilled water to remove the residual acid. The samples were then dried. Activation of the Samples were done at 900 °C.

RESULTS & DISCUSSION

Initially the surface area of carbon black obtained was approx 86.855 m²/gm. This value is close to the average value of surface area of commercial carbon black used in passenger car tyre such as N330 (A=82 m²/gm) and N339

(A=90 m²/gm). Other authors have also reported the similar values 9-11.

The samples were prepared by acid demineralization and KOH Treatment. The various tests listed below were done for the final results obtained from the samples.

1. BET(Brunauer–Emmett–Teller):

Untreated Carbon Black

Slope: 39.252
Intercept: 8.442e-01
Correlation coefficient, r: 0.999996 C
Constant: 47.498

Surface Area: **86.855 m²/g**

HCl Treated Carbon Black 1M

Slope: 76.593
Intercept: 3.115e+00
Correlation coefficient, r: 0.999709 C
Constant: 25.587

Surface Area: **43.692 m²/g**

HCl Treated Carbon Black 5M

Slope: 69.913
Intercept: 5.092e+00
Correlation coefficient, r: 0.996259 C
constant: 14.731

Surface Area: **46.432 m²/g**

HCl Treated Carbon Black 10M

Slope: 82.680
Intercept: 3.254e+00
Correlation coefficient, r: 0.999835 C
constant: 26.412

Surface Area: **40.526 m²/g**

KOH treated and Activated

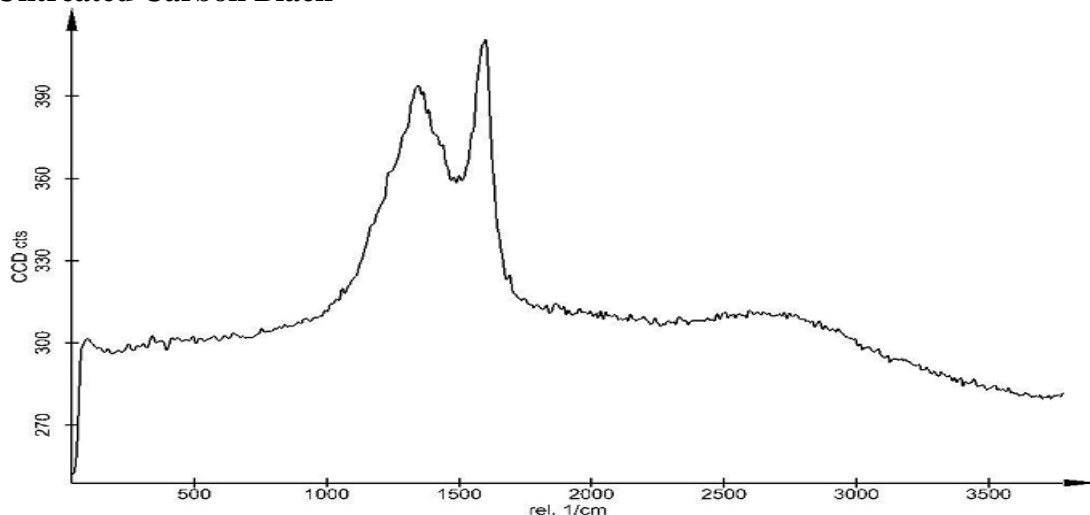
Slope: 7.869
Intercept: 5.033e-02
Correlation coefficient, r: 0.999526 C
constant: 157.366

Surface Area: **439.746 m²/g**

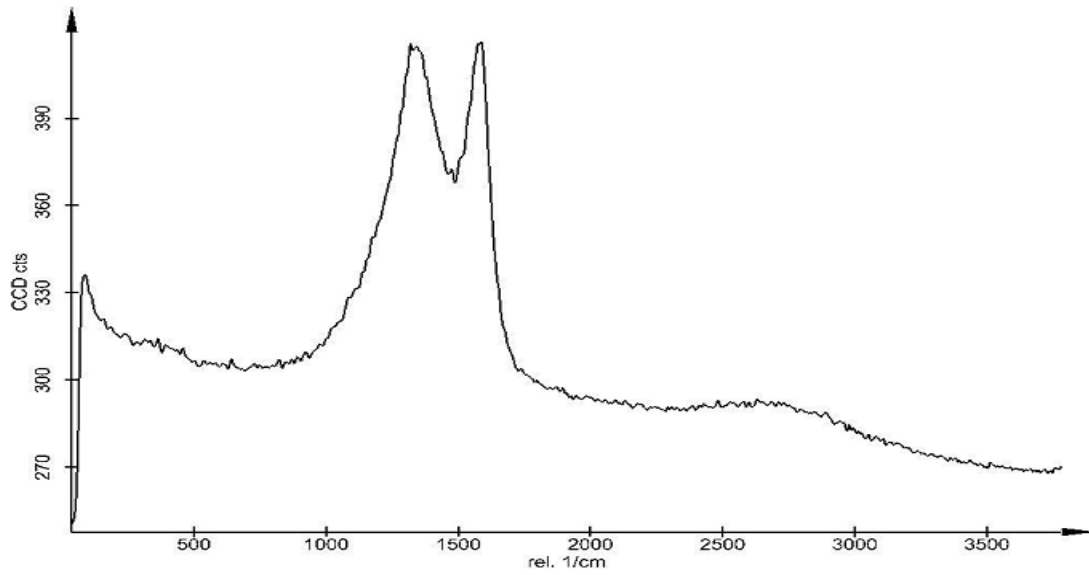
□ **BET Report:** The surface area obtained from Brunauer–Emmett–Teller (BET) theory showed major difference between before and after HCl treatment. Surface area almost got halved after HCl treatment. But after KOH treatment surface area increased tremendously. The surface area of the untreated carbon black is very close to the average value of surface areas of commercial carbon blacks used in passenger car tyres. But so much change in surface area suggests that the structure of carbon black obtained from tire recycle is different from commercial carbon black.

2. RAMAN TEST:

Untreated Carbon Black



Carbon Black HCL treated



Raman test report: After HCl treatment the carbon black became a little more crystalline as 3. EDEX Test:

the intensity of D band increased slightly while the G band remained almost the same.

Untreated carbon black:

Element	App	Intensity	Weight%	Weight%	Atomic%
	Conc.	Corrn.		Sigma	
C K	13.45	0.9174	78.15	1.47	87.20
O K	0.98	0.4955	10.57	1.36	8.85
Al K	0.12	0.9760	0.65	0.19	0.32
Si K	0.46	0.9915	2.46	0.28	1.17
S K	0.51	0.9618	2.81	0.33	1.17
Cl K	0.16	0.8049	1.05	0.24	0.40
Zn L	0.48	0.5900	4.31	0.58	0.88

Carbon black 1M:

Element	Weight%	Atomic%
C K	85.57	92.66
O K	5.83	4.74
Si K	1.14	0.53
S K	2.86	1.16
Zn L	4.61	0.92
Totals	100.00	

Carbon black 5M:

Element	Weight%	Atomic%
C K	88.09	93.45
O K	5.45	4.34
Si K	1.94	0.88
S K	2.19	0.87
Zn L	2.33	0.45
Totals	100.00	

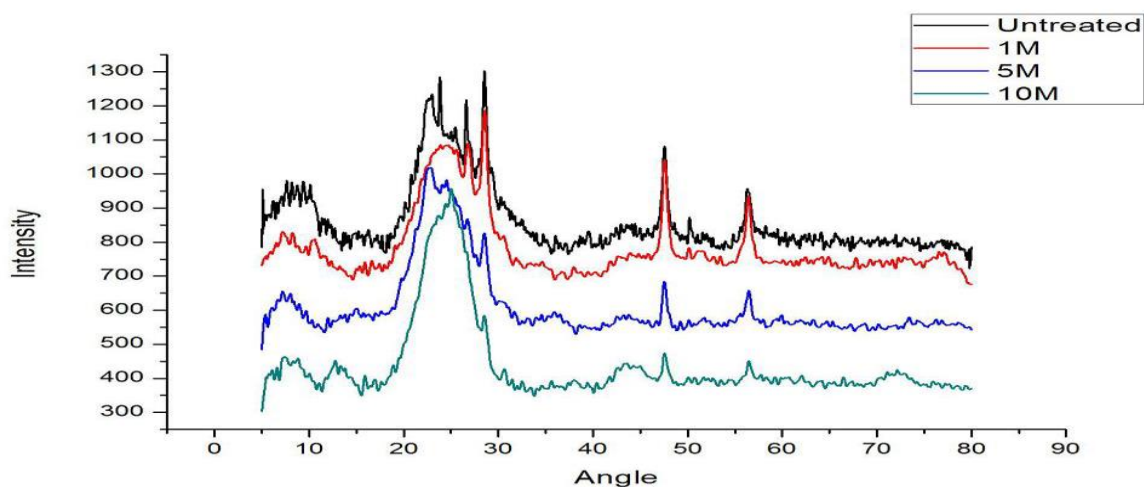
Carbon Black 10M:

Element	Weight%	Atomic%
C K	89.76	93.90
O K	5.70	4.48
Si K	1.46	0.65
S K	1.87	0.73
Zn L	1.21	0.23
Totals	100.00	

EDEX Test Report: Before HCl treatment carbon black had some major impurities such as Oxygen, Sulphur, Silicon and Zinc and after HCl

demineralization these impurities decreased but there was no significant change w.r.t. molar concentration of HCl.

4. XRD Test:



XRD Test Report: The graph the peak at 25 $^{\circ}$ gets sharper and sharper so we can say that the material becomes more crystalline after HCl treatment and with higher molar conc. it becomes more and more crystalline.

CONCLUSION:

A carbon black was produced from waste tyre rubber through thermal pyrolysis and demineralized with acid prior to activation. Acid treatment is an efficient way to demineralize and increase the surface area of the carbon black after activation at 900 C. This reveals that the acid treated carbon black prepared from waste tyres is suitable. The surface area enhancement and the relatively high apparent activation energies suggest that gasification under these conditions is approximately chemically controlled. The characteristics of the prepared carbons, in terms of porosity and surface area, depend on the activation degree but also on the nature of the activating agent and in a lesser extent, on the process temperature.

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