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Research Article

Acid Base Demineralization of Pyrolytic Carbon Black Obtained From Waste Rubber

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Abstract

Ash content reduction in pyrolytic carbon black obtained from waste rubber is investigated in current work using acid demineralization technique followed by base demineralization. A number of acid samples of different concentrations (50% HNO_3, Aqua Regia, Aqua Regia+H_2SO_4 (2:1), 50% HCl + H_2SO_4) have been used whereas for basic demineralization of some samples, sodium Hydroxide NaOH is used. The objective of the study was the acid base demineralization to enhance activation of carbon.

Keywords: Pyroliysis; Scrap Rubber Tyres; Acid-Base Demineralization; Pyrolytic Carbon; Activation Number; Iodine Number

Introduction

Waste rubber tyres are a problem disposal for decades, they do not degrade easily. A large amount of tyres in dumps and landfills are accidental fire and environmental hazard together with that they are a friendly site for breeding of insects and flies. Environmental safe and feasible new recycling technologies are required. There are major aspects of tyre problems such as tyres stockpiles provide breeding ground for mosquitoes and vermin, this in turn, causes serious diseases and affects human health. Fire hazards in 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. Waste rubber tyres are rich in hydrocarbons and are a potential source of energy, fuel and raw materials. Over the past few years pyrolysis of scrap tyres with recovery of chars, oils and gases are considered economically promising and environmentally acceptable [1].

The solid residue of pyrolysis of scrap tyres are mixture of carbon black, high content of ash and coke. The ash generally consists of zinc sulfide, zinc oxide, silica, alumina etc. The carbonaceous deposits and inorganic matter covers active sites and hinders the recycling of pyrolytic carbon.

In this study different acids are used to study the removal of ash contents and increase of active sites of carbon sample, further more different mixtures of acids were used to study the effect on activation of carbon.

Methods and Materials

Materials

The chemicals used in this work are Pyrolytic carbon black, which was provided by a local company (About 200g of tyre shreds of equal size were set in a tube furnace at 900°C was heated at atmospheric pressure solid residue was collected and ground (Figure 1), mixtures of different compositions of Hydrochloric acid (HCl), Nitric acid (HNO_3), Sulphuric acid (H_2SO_4) and sodium hydro-oxide (NaOH) were prepared at ICET [2].

Experimental Setup

Pre-Treatment

i. Magnetic Separation: Magnetic separation of powdered Carbon Black sample was done to remove the iron particles present in the sample. 200g of the powdered sample was taken. A bar magnet was covered with a plastic sheet and then sifted through the sample thoroughly. Iron fillings got attached to it which was then removed from the magnet and same procedure was repeated twice until all the iron fillings were removed from the Carbon Black sample. It is necessary to remove the Iron particles because they can cause contamination of the sample and can cause formation of surplus chemical compounds during treatment with acids and base.

ii. Water Washing: After Magnetic separation the sample was washed with distilled water. It was done to remove soluble impurities from the sample. Water does not affect the properties of Carbon Black as it is immiscible with water. The sample was then filtered using Whatman No. 42 filter paper and dried in hot air oven for 24hours [3].

Acid demineralization

An acid demineralization process was carried out to decrease the inorganic impurities as well as to remove un-desirable (ash) contents

Table 1: Experimental calculations of iodine value of the activated carbon.

Material	Vol A	Vol B	C=B-A	Iodine#
	ml	ml	ml	mg/g
100% HCl + NaOH	18.2	21	2.79	423
Aqua regia	17.5	21	3.5	528
50% HCl + H_2SO_4	16	21	5	755.5
50% HNO_3 + NaOH	15.6	21	5.4	815.9
Aqua regia +H_2SO_4 (2:1)	15.3	21	5.7	861.2
50% HNO_3	14	21	6.2	936.8
50% HNO_3 + H_2SO_4+ NaOH	13.5	21	7.5	1133.2
100% HNO_3 + NaOH	13.2	21	7.8	1178.5
Aqua regia + NaOH	12.7	21	8.3	1254.1

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from the sample. Hydrochloric acid (HCl), Nitric acid (HNO_3) Sulphuric acid (H_2SO_4) and there mixtures were selected for the acid demineralization. Solutions and mixtures of different concentrations of these acids were prepared and demineralization was conducted at room temperature. Known amounts of the Carbon Black sample were taken and put in the beakers containing the acid solutions, samples were stirred for 20 minutes at room temperature, and then they were left immersed in acid to be demineralized for a period of 24h, a length of time that was considered to be long enough not to limit the demineralization process. Whattman 42 filters paper and thoroughly rinsed with distilled water to remove the residual acid [4]. This procedure was repeated for all the acids and different samples were made.

Basic demineralization

After the acid demineralization, basic demineralization was conducted on the washed and dried acid treated samples. They were treated with 1N Sodium hydroxide (NaOH) and heated for 15Minutes with constant stirring the solution was diluted with water and left for few hours. Care was taken not to leave the samples immersed in base for too long (more than 24h) as it causes ingress of moisture into carbon pores. The base treated samples were then filtered, washed with distilled water and dried at 110°C for 24h in the oven [5]. Synopsis scheme of Acid-Base treatment has been shown in the Figure 2.

Activation tests

Out of many testing techniques for testing of activity of pyroytic carbon, following two techniques were used in order to check the increase in activation of carbon samples.

- Iodine Value Test
- Oxalic Acid Test

Iodine Value Test: The determination of the iodine number is a simple and quick test, giving an indication of the internal surface area of activated carbon. Iodine Number; "The number of milligrams of iodine adsorbed from an aqueous solution by 1mg by 1g of activated carbon when the iodine concentration of the residual filtrate is 0.1N."

0.2g of treated carbon samples were taken and mixed in 0.1N Iodine solution (40 gm KI and 12.7 g of I2 in 1L of Distilled water) for 20 minutes then it was titrated against 0.1N Sodium Thiosulphate solution. The results obtained are shown through Table 1.

Iodine value = C x Conversion factor (i)

C = B-A

B = Burette reading corresponds to blank reading.

Table 2: Experimental data of volume of Oxalic Acid used, indicating degree of activation.

Material	Vol of Oxalic Acid Used	Degree of Activity
	ml	*10^3 / ml
Raw Sample	17	58.8
100% HCl + NaOH	15.8	63.3
Aqua Regia	15.5	64.5
50% HCl + H_2SO_4	14.7	68
50% HNO_3 + NaOH	14.5	68.9
aqua Regia +H_2SO_4 (2:1)	14.3	69.9
50% HNO_3	14.8	67.5
50% HNO_3 + H_2SO_4+ NaOH	14.2	70.4
100% HNO_3 + NaOH	13.9	71.9
Aqua Regia + NaOH	13.5	74

A = Burette reading corresponds to Activated Samples.

Conversion Factor = (Mol wt. of iodine (127) x normality of iodine 40) (ii)

(Wt. of carbon x Blank reading)

Oxalic acid test

Procedure: 0.1N Oxalic acid solution was prepared by adding 12.6g of oxalic acid in 1000ml of water. 0.2g of treated carbon samples was taken and 40ml of 0.1N Oxalic acid solution was added to them. The mixture was stirred continuously for 30 minutes. The treated solution was filtered and 10 ml of it was taken for titration against 0.1N NaOH after adding phenolphthalein indicator. End point was observed when the solution turned pink. The amount of base used was noted and reported as shown in Table 2.

Results and discussion

The test results have shown that carbon obtained from waste rubber can be activated by reducing ash content to a great extent using acid and base demineralization method in mild conditions of temperature and pressure. Table l, summarizes the results of Iodine number indicating degree of activation for the pre-treated and post-treated sample of Carbon Black. Proximate analysis of pre-treated samples indicate that the rubber had a typical volatile content of 2.9%, fixed carbon of 85.15%, ash content of 11.7% and moisture content of 0.25%. The treatment of 100 % HNO_3 and NaOH with Carbon Black increases activation degree upto~3X while treatment with Aqua Regia and NaOH will result in increase of activation degree up to 3.2X. This is because of Chlorides and Nitrates formed in the presence of HCl, $HNO_3$and NaOH. Maximum increase in activation i.e. reduction in

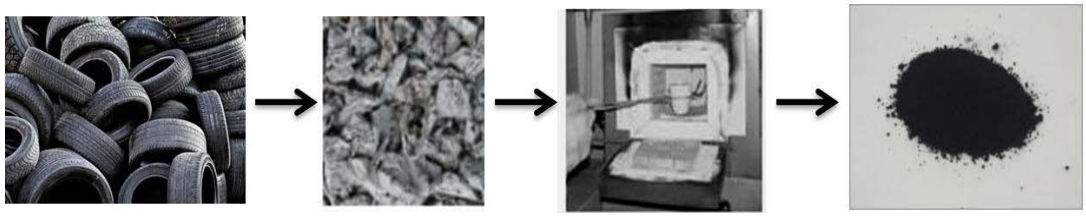

Figure 1: Diagram for pyrolytic and thermal shock processes.

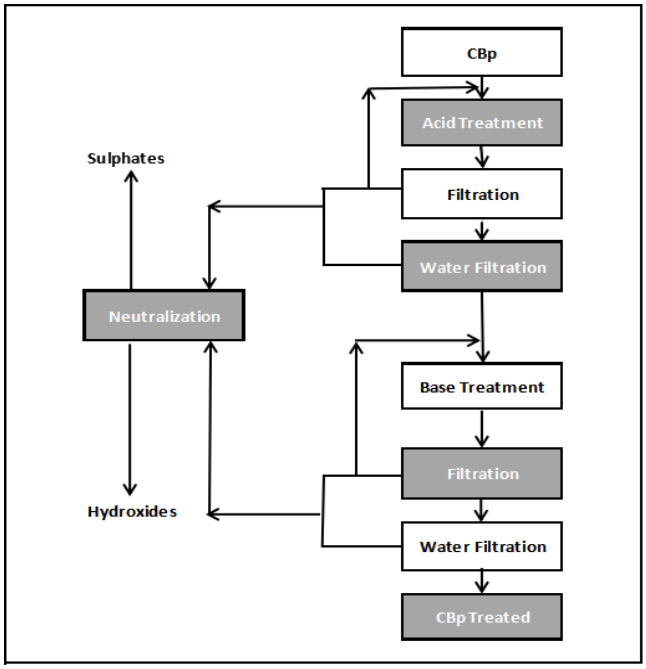

Figure 2: Synopsis scheme of Acid-Base treatment.

Ash content was observed by treating pyrolytic carbon black sample with Aqua Regia and NaOH in 1:1 ratio.

Conclusion

Analysis of the results obtained, shows that the acid-base treatment is an efficient way to decrease the ash content of the pyrolytic carbon black obtained by vacuum pyrolysis of used tires. Reducing the ash content increases the surface area of the carbon black particles and expands the range of utilization of the CBp i.e Figure 3. Maximum

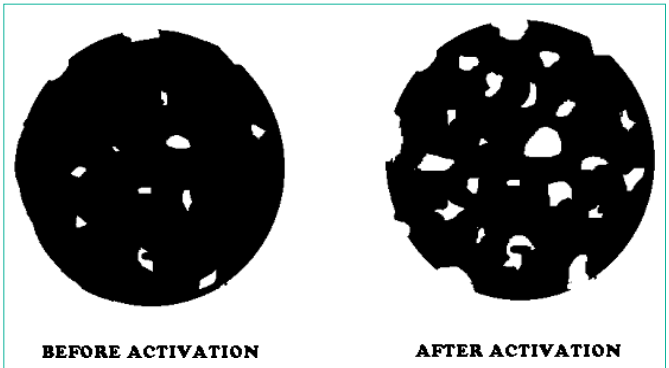

Figure 3: Two-dimensional representation of carbon activation.

activation was observed by using HNO_3 and NaOH in 1:1. By varying the concentration of acid and base samples, results may be varied. The soluble and non-soluble salts formed (sulphates and hydroxides, respectively) by mixing the spent sulfuric acid and sodium hydroxide can be used for other applications.

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