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Synthesis of Coconut Shell derived Activated Carbon NBR Composite for Automobile Application

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Abstract - In automobile sector, PVC/NBR rubber blend is filled with activated carbon to prepare fuel tube . Activated carbon alters surface properties.,Because Acid is used to activate the carbon. This acid treatment develops reaction sites on carbon surface and increases compatibility of carbon with rubber. To study this, activated carbon derived from lignocellulosic material *viz*: coconut shell is used.The carbon is activated by phosphoric acid as dehydrating agent in the stoichiometric ratio of 3:1.Activated carbon found comparable with commercial carbon Petrol swelling test ASTM standard D-471-98 (€) results, shows the least deviation in hardness and mechanical propertiesin comparison with commercial rubber. The composite prepared are of 20-Parts per hundred of rubber (PHR) basis. The results obtained are correlated with reference to proximate analysis and hence concluded , fuel tube can be prepared from coconut shell carbon NBR composite.

Index terms**:** Lignocellulose, phosphoric acid, Activated carbon, nitrile butydiene rubber, Composites, D-471-98 (€)

I . Introduction

Lignocellulose refers to the specific structure of biomass. The main constituents of lignocellulosic biomass comprise lignin, hemicellulose and cellulose. This is a complex structure in which the cellulose is surrounded by a monolayer of hemicellulose and embedded in a matrix of hemicellulose and lignin. Esters and ethers are the integral parts of the lignocelluloses. Also, carbonyl groups are present over three of the carbohydrates, namely lignin, cellulose and hemicelluloses. This structure is as shown in figure 1.

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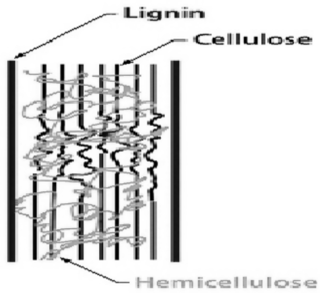

Fig 1: Structural representation of lignocellulose

Activated carbon is a versatile material with high porosity and surface area. The activation may be physical or chemical. Chemical activation reduces tar formation. Chemical activation is carried out with wood as the starting material. In the beginning, wood is impregnated with a concentrated solution of activating agents. It results in degradation of cellulosic material. Chemical-impregnated material is then pyrolysed at 400^0C in the absence of air. Pyrolysed product is cooled and washed to remove activating agent, which is recycled. On calcination, impregnated and chemically dehydrated raw material results in charring and aromatization, and creation of porous structure. Pottasium disulphide, alkali metal hydroxide, and carbonate and chlorides of $Ca+^2$; Mg^{+2} and Fe^{+3}. Activating agent acts like dehydrating agent which influences the pyrolytic decomposition and inhibit the formation of tar. Also, it decreases the formation of acetic acid, methanol etc. and enhance the yield of carbon. Due to enhanced surface properties, it has become one of the technically important material for selective separations commercially. Nevertheless, its application fields are restricted due to high cost.

This difficulty has led to search for the use of cheap and efficient alternative materials such as rice husk [1], bamboo [2], sugarcane stalks [3], tamarind kernel powder [4], palm shell [5] babool wood [6], bagasse, fly ash [7], ashoka leaf powder [8], coir pith [9] and banana pith [10] etc. Biomass wastes are considered to be a very important feedstock because they are renewable sources. Activated carbon such produced can be used as effective adsorbent because of high adsorptive capacity. However, continuous production of activated carbon with reproducibility of characteristics is restricted by the seasonal availability of the starting material.

To address this issue partly, Coconut shell which is available throughout the year irrespective of season, is used as starting materials for carbon preparation. Rubber industry is a huge consumer of carbon as reinforcing material. The filler is used to impart certain properties to the rubber material such as colour, hardness and strength up to certain extent. For activation, the surface of carbon modifies to develop newer reactive cites inclusive of ester, ether and carboxylic.

NBR is a complex family of workhorse elastomers. The unique balance of oil, chemical, heat and cold resistance allows it to work well in a wide variety of automotive and industrial applications. The nitrile rubber, is used for study. Russian raw rubber normally used to blend with PVC for automobile applications.

Nitrile rubber $-(CH_2\text{-}CH{=}CH\text{-}CH_2)-$ is generally resistant to aliphatic hydrocarbons. When compounded with activated carbon, the ester and ether linkages present on activated carbon may entangle with zinc present in rubber improves physical properties.

II. Materials and Methods

A Precursor for Activated Carbon

Dry coconut shell samples were procured from coastal Karnataka, India. The fibrous portion of the shell was cut into small (10 mm x 10 mm) size pieces and stored.

B. Preparation of activated carbon

First preparation of activated carbon was done in three batch sizes of 50 gm, 100 gm, and 300 gm. H_3PO_4 chemically pure quality [Merck and Co.] was used as activating agent. A known mass of activated agent was mixed with distilled water, and Biomass waste was impregnated in acidic solution. The amount of phosphoric acid solution used was adjusted to give a certain impregnation ratio (weight of activating agent/weight of raw material) of 3:1 . The impregnated sample was kept for 24hr. After 24hr the residual water was removed and kept in oven for 110^0C A weighed amount of impregnated samples was kept in muffle furnace for 400^0C. The muffle furnace is purged with high purity nitrogen gas to avoid oxidation .Nitrogen flow was adjusted to 3ml/^{0}c at 400^0C. The activated carbon was subsequently removed from furnace and cooled to room temperature.

After activation the samples, 3M hydrochloric acid used to remove the phosphoric acid compounds. The washed samples were dried at 110^0C for 6hr in oven and then ground to form a porous carbon powder. The equipment was fabricated to hold the raw sample of 30 * 9 * 9 cm size as shown in figure 2.

Fig 2: Fabricated Equipment to hold the sample

Fig 3: Experimental Set Up

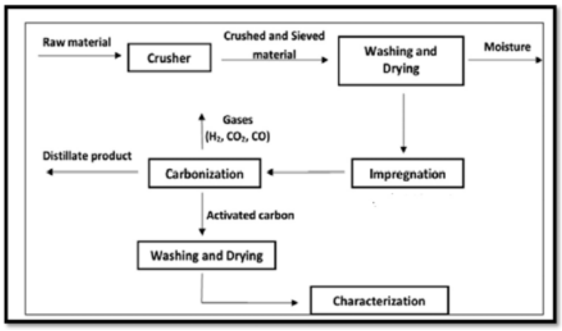

Fig 4: Schematic diagram for preparation of activated carbon from coconut shell

C. Scanning Electron Microscope (SEM) Analysis

The prepared activated carbons were examined by Scanning Electron Microscope (SEM) to analyze the surface of the adsorbents. SEM micrographs of the chemically activated carbons by H_3PO_4 are presented in Figure 5. Well-developed porous surface was observed at higher magnification. The pores observed from SEM images are having diameter in micrometer (µm) range. These pores are considered as channels to the microporous network. From the figures below, it can be observed that the adsorbent have rough texture with heterogeneous surface and a variety of randomly distributed pore size.

D. Proximate and Ultimate analysis of activated carbons

Please refer to table 2.

III. Results and Discussion

Lignocellulose content of the starting material

From Table 1, it is depicted that the acid hydrolysable is more in coconut shell. Coconut shell is the material which has combination of fibrous and hard material. The same trend is observed in ultimate analysis results. The % yield of carbonisation also follows the same trend as shown in above analysis. minutes with 175^0C to 190^0C in less than 1 minute accelerated heating rate. During this process the material is subjected to undergo mechanical force at elevated temperatures necessary for the processing of the material to cast sheet at compression moulding machine.

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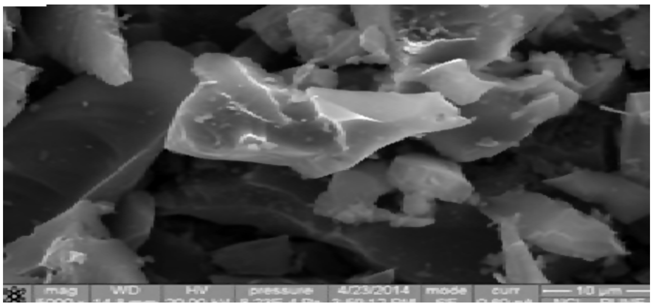

Fig 5 : SEM images of chemically activated adsorbent, for 3:1 Acid: Adsorbent ratio. (A) Coconut Shell

FT-IR analysis

To investigate the functional groups of the activated carbon generated by chemical activation of three of the lignocelluloses, a FT-IR study was carried out and the spectra are shown in Figure 5. The spectra shows prominent absorption peaks, in common, corresponding to carbonyl C=O stretching @ 1670 to 1820 cm^{-1}, ether. The raw materials used are the fibre based. The major component for this type of materials is cellulose bound together by lignin. Hemicellulose is also present over the material. Hydroxyl (-OH) functional group tend to undergo esterification reaction in the presence of acids. For, the activation by phosphoric acid was followed at room temperature, generated the esters over the material. Otherwise the material contains ether linkages prominently. Thus produced ester linkages undergo ionic bonding with nitrile rubber when processed over two roll mill. The test results are depicted in Table 3. Here TS1 **st**ands for torque generated at the initiation of the curing of the material. TS2 indicates torque during curing of the material and TS3 torque for complete curing of the material.

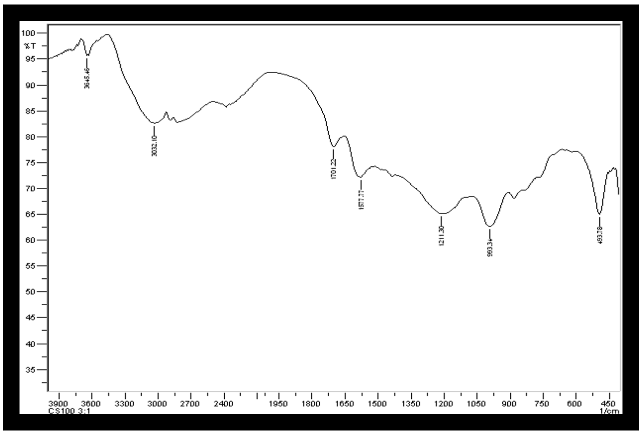

Fig 6 : FTIR for activated carbon prepared from coconut nut shell

Activated carbon give rise to reinforcing effect and therefore the aggregate is smallest form of a given activated carbon grade well dispersed in an elastomer, that will still keep all the reinforcing capabilities of a filler same. Activated carbon aggregates contain internal voids which are capable of absorbing polymer. NBR Coconut shell Activated carbon composite is as shown in figure 7.

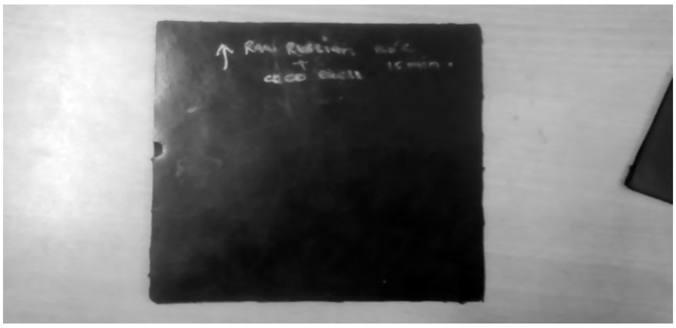

Fig 7: NBR- Coconut Shell activated carbon Composite

Rheology of rubber blend

As standard practice, to find out the processing condition the blends were tested for rheology using moving die rheometer. The rheometer used was complies with ASTM D5289, ISO 6502 GB T/16584. Heating rate of the rheometer was 30^0C to 190^0C in less than 6.

Petrol Swelling Test (ASTM Standard no - D-471-98)

To assess the extent of swelling behaviour of composites, Petrol Swelling test ASTM Standard no - D-471-98[18] was carried. Following are the trends observed in the results. This test provided information on the interface strength, degree of dispersion of carbon and their alignment in the elastomeric matrix. Following results show the swelling index in terms of after ageing and before ageing. Petrol swelling test is also predicted in terms of deviation of density

and hardness. From Fig 8-9 predict coconut shell based activated carbon show less deviation for the petrol soaking.

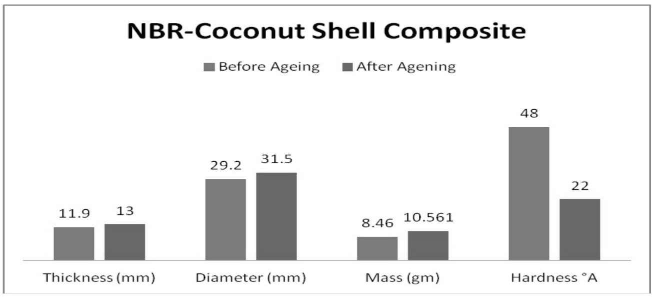

Fig.8 : Coconut nut- NBR composite

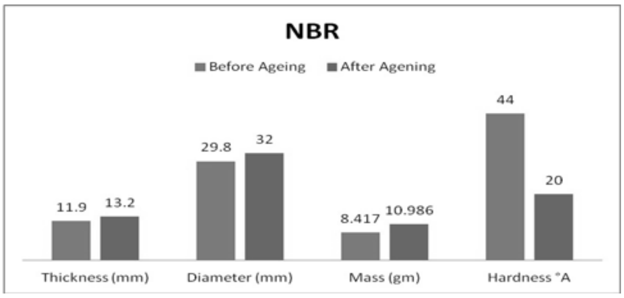

Fig. 9: NBR composite

Thus material was found comparable with commercial carbon based composite. The fuel pipe or hose pipe application was aimed and the composite with commercial matrix were prepared. NBR -Coconut shell composite shows least deviation in hardness for petrol soaking but the maximum deviation in density. Please refer to table 4.

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IV. Future Scope

The chemistry behind probable entanglement of activated carbon with NBR is to be studied further. As the NBR (raw) based activated carbon composites were comparable to the commercial carbon based NBR (raw) composites, To know the usability of activated carbon on industrial level, this coconut shell activated carbon is proposed to use as filler from 20 to 70 phr in commercial blend Elastorene 673 EL (70/30 NBR: PVC, W/W) formulation.

V. Conclusion

The results of this study showed that coconut shell can be successfully converted into activated carbon by using H_3PO_4 as dehydrating agent. The activated carbon has developed ether, ester and carbonyl groups over the surface for H_3PO_4 treatment. The reaction mechanism is proposed for the entanglement of activated carbon with rubber by the formation of ionic bond. H_3PO_4 used for chemical activation has different impact on surface conversion of lignocelluloses pertaining to the lignin content of starting material. The coconut shell carbon found comparable with commercial carbon in formation of rubber based composite for automobile applications.

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Table 1: Lignin and cellulose content of the raw material

Sr. No.	Material	Cellulose % (as acid soluble)	Lignin % (as alkali soluble)	Weight of remainder
03	Coconut shell	58%	16%	26%

Table 2: Proximate and Ultimate Analysis of activated carbons

Activated carbon (wt%) dry basis	
Parameters	Coconut Shell
Proximate Analysis	
Moisture	9.93
Ash	4.5
Fixed Carbon	85.44
Volatile Matter	3.99

Activated carbon (wt%) dry basis	
Parameters	Coconut Shell
Ultimate Analysis	
Carbon	59.62
Hydrogen	2.43
Nitogen	0.12
Sulphur	0.1

Table 3: Rheological behaviour of the samples

Sr. No.	Material	TS 1 (N-m)	TS 2 (N-m)	TS 3 (N-m)
01	Coconut shell	1.21	1.45	1.89

Table 4: Effect of petrol swelling on NBR (raw) activated composites

Sr. No	Sample	% Deviation in density	% Deviation in hardness	Remark
1	NBR	87.18	54.16	This is matrix material
2	CS-NBR	28.3	54	This can be used for hose pipe application