Comparative Studies on Effect of Activated Carbon from Different Sources as Filler in NBR for Automobile Application

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Abstract: **This study reports usability of activated carbon obtained from areca nut shell, coconut shell, and coconut leaves as filler to prepare NBR based composite for automobile based application. The carbon is activated by H3PO4 as dehydrating agent in the ratio of 1:1 for 300g batch size. It is found that in comparison with commercially available carbon filler, the prepared carbon responded better to the petrol swelling test. Amongst the three sources namely, areca nut shell, coconut shell, and coconut leaves, the carbon obtained from coconut shell source appeared better as far as percent swelling and percent deviation in degree of hardness is concerned. The composite prepared are of 20PHR basis. The results obtained are tried to correlate with reference to proximate analysis.**

Keywords: **Automobile application, Activated carbon, NBR, Composites, Shore -A**

I. INTRODUCTION

Activated carbon is an extremely versatile material with high porosity and surface area. It has become one of the technically important materials for selective separations. The structure of activated carbon is mainly micro porous. 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. Activate carbon such produced can be used as effective adsorbent because of high adsorptive capacity. However, continues production of activated carbon with reproducibility of characteristics is restricted by the seasonal availability of the starting materials.

This led us to use coconut shell, areca nut shell and coconut leaves, which are available throughout the year irrespective of season as starting materials for carbon preparation. Being in daily use in Indian continent as food ingredients and in some parts on auspicious occasions, the coconut shell is generated enormously. Areca nut is also chewed in Asian continent as mouth freshener and in India on auspicious occasions along with coconut.

Rubber industry is a huge consumer of carbon as reinforcing material. The filler, to impart certain properties to the rubber material such as colour, hardness and strength up to certain extent. However, so far there is no report for the use of activated carbon as filler material in rubber. For activation, the surface of carbon modifies to develop newer reactive cites. There are different agents used for activation of carbon. Each agent imparts different morphology to the resultant carbon. The ratio of lignin to cellulose and the nature of starting material play certain role in the attribution of activity to the resultant carbon. For the treatment with alkali, the surface modification is limited. The acid treatment enhances porosity, and in turn increases the yield of activated carbon. Ours is the activated carbon prepared by acid treatment to the agricultural waste lignocelluloses materials.

Polymer based composite materials have been used widely in home appliances, construction, automotive industry, packaging application, aircraft engine blades due to their excellent specific mechanical and tribological properties for hundreds of years by Guadagno et al. [11].

In this work we are reporting, probably for the first time, the use of activated carbon as filler in rubber industry. The idea behind incorporation of activated carbon is chemical reaction between nitrile rubber and the surface modified carbon imparted with ether, ester linkages on the surface.

In present work various properties of carbon composite prepared from coconut shell, coconut leaves and areca nut shell were compared with commercial carbon composite to check the feasibility for commercial purpose and its application in automobile industry.

II. MATERIALS AND METHODS

2.1 Selection of materials: By finding out the acid soluble and alkali soluble content of the different agricultural wastes, the coconut and the areca nut material were selected pertaining to the availability and the acid hydrolysable content.

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Biomass waste such as coconut shell, areca nut shell and coconut leaves is used as the raw material for preparation of activated carbon. The biomass is first chopped into pieces of 2 cm wide and 5 cm long. Then washed with distilled water to remove dust particles, and then dried at 110^0c. Biomass waste was finally crushed and sieved to 180 mesh size.

Methods -Experimental

2.2 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 mass ratio of activating agent to dried material was 1-3.

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:-1. The Experimental set up of preparation of activated carbon from coconut shell, areca nut shell, and coconut leaves consists of muffle furnace associated with nitrogen gas cylinder is shown in figure no:2

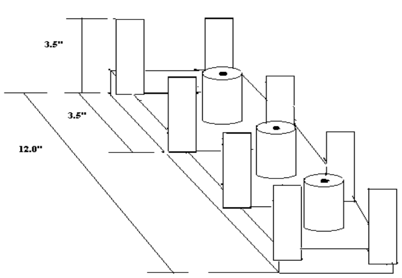

Figure no:-1 Fabricated Equipment to hold the sample

2.3 Analysis of starting material: In order to find out the cellulose content as acid hydrolysable and lignin content as alkali soluble, 73% H_2SO_4 solution and 17% NaOH solutions were used. The pre-weighed sample was first digested in acid at room temperature for 4hrs with constant stirring. Oven dried to constant weight to know the weight difference. The weight difference corresponds to the cellulose and hemicelluloses content. The same sample was digested in NaOH solution at 80^0C for 2 hrs with constant stirring. The sample was oven dried till constant weight and weighed to find the lignin as alkali soluble content.

2.4 Analysis of activated carbon: The Ultimate Analysis of a sample determines the elemental composition of the sample. It is based on the principle of Dumas method which involves the complete and instantaneous oxidation of the sample by flash combustion. The results are in percentage composition of Carbon, Hydrogen, Nitrogen and Sulphur. From these results the oxygen composition is determined by subtracting the sum of Carbon, Hydrogen, Nitrogen, and Sulphur compositions from 100.

2.5 Preparation of Carbon Composite: The compounding of composites is done as reported in Table 1. Rubber chemical such as ZnO, Sulphur and stearic acid were supplied by Sunrise Elastomers, MIDC, Mhallunge, Pune. The blending was carried out using two roll-mill at room temperature. The slabs of 10×10×0.5 cm were developed using compression moulding machine at 140^0C under pressure of 1Mpa for 12 minutes with reference to the rheology results of the material.

The mechanical and tensile properties were calculated using tensile testing machine and hardness tester supplied by 'Microvision Enterprises' Haryana. The composite prepared in the form of slab 10×10×0.2 cm was cut in dumb-bell shape as per ASTM Standard no -D -412-98 for tensile testing. The same sample was used for hardness testing.

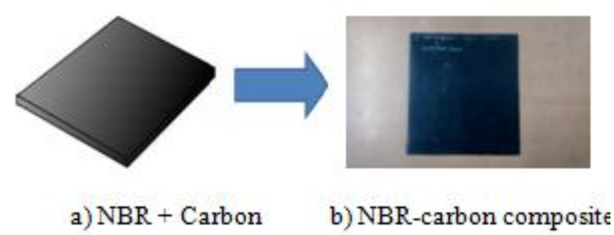

Fig no: 2 NBR Composite

Table 1 Composition of Material for different biomass wastes

Arecanut Shell		Coconut leaves		Coconut Shell	
Material	PHR*	Material	PHR	Material	PHR
NBR	100	NBR	100	NBR	100
ZnO	5	ZnO	5	ZnO	5
Stearic Acid	1	Stearic Acid	1	Stearic Acid	1
Sulphur	1.5	Sulphur	1.5	Sulphur	1.5
MBT	1.5	MBT	1.5	MBT	1.5
Carbon	20	Carbon	20	Carbon	20

PHR* -Parts per hundred of rubber

2.6 Petrol Swelling Behaviour of rubber composite: In order to study the response of formed rubber composite using various filler materials, to the petrol for its application in

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automobile, the composite prepared was tested as per ASTM standard D-471-98 (€).

2.7 Tensile Testing of rubber composite In order to study the tensile property of formed rubber composite using various filler materials, for its application in automobile, the composite prepared was tested as per ASTM standard D-412-98 a reapproved 2002 (€).

III. RESULTS AND DISCUSSION

3.1 Lignocellulose content of the starting material

From the Table no. – 2, it is observed that the acid hydrolysable is more in areca nut shell as compared to the other two materials. Three of the materials present three different classes of lignocellulosics. Coconut shell is the only woody material i.e. more complex and cross linked form of lignocelluloses. Areca nut shell is the material which has combination of fibrous and hard material. Whereas coconut leaves has on major watery content, comparatively simple form of polymer lignocelluloses.

The lignin and cellulose content varies from species to species of the plants. Also, in general the cellulose content is observed to be greater than that in non-dried conditions. It is due to the water portion; that contributes neither to the lignin nor to the cellulose vaporises and too the cellulose and lignin content are expressed on dry weight basis [12].

Table –2: Lignin and cellulose content of the raw material

Sr. No.	Material	Cellulose % (as acid soluble)	Lignin % (as alkali soluble)	Weight of remainder
01	Coco nut shell	36%	16%	48%
02	Coco nut leaves	47%	40%	13%
03	Areca nut shell	58%	16%	26%

The same trend is observed in ultimate analysis results. The coconut shell has got all the value, for ultimate analysis in moderate range as compare areca nut shell and coconut leaves except the % fix carbon content. Which is the highest i.e 81% followed by areca nut shell 75.9% and coconut leaves 75.3%.

The % yield of carbonisation also follows the same trend as shown in above analysis

3.2 Elemental Analysis

The Ultimate Analysis was carried out in a CHNS Analyzer. The sample is fed into the analyzer along with excess supply of oxygen. The reaction of oxygen with other elements (namely carbon, hydrogen, nitrogen, and sulphur, present in the sample, produces carbon dioxide, water, nitrogen dioxide, and sulphur dioxide respectively. The combustion products are separated by a chromatographic column and are detected by the thermal conductivity detector (T.C.D.), which gives an output signal proportional to the concentration of the individual components of the mixture. This determines the equivalent compositions of elements in the sample.

Table 3 presents the values in the terms of Lignocellulosic wastes (wt %) dry basis.

Table no: 3 Ultimate Analysis Results

Parameters	**Lignocellulosic wastes (wt%) dry basis**		
	Areca nut Shell	**Coconut leaves**	**Coconut Shell**
Proximate Analysis			
Moisture	15.46	8.167	11.93
Ash	6.473	2.39	4.90
Fixed Carbon	75.9	75.38	81.44
Volatile Matter	6.47	14.06	4.985
Ultimate Analysis			
Carbon	54.86	23.56	57.62
Hydrogen	4.486	0.916	3.432
Nitrogen	0.475	0.889	0.191
Sulphur	0.251	0.115	0.119

3.3 Petrol Swelling Test

In order to assess the extent of swelling behaviour of composites, Petrol Swelling test ASTM Standard no - D-471-98 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. 3 – 6, it is clear that the coconut shell based activated carbon filled and areca nut shell based activated carbon filled rubber composites show less deviation for the petrol soaking. The coconut leaves based activated carbon filled rubber composite show maximum density deviation. This is because of the maximum portion of lignocelluloses in leaves is water soluble sugars.

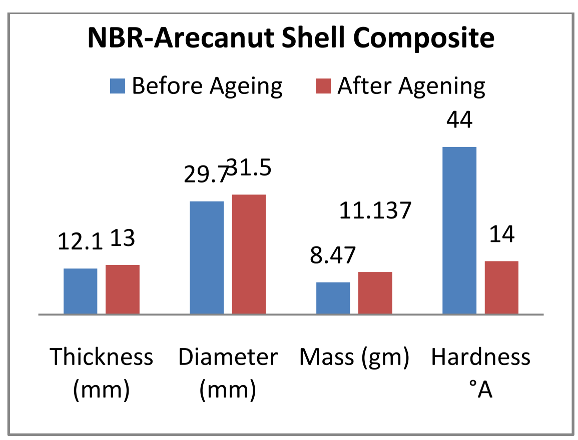

Fig :3 Arecanut- NBR composite

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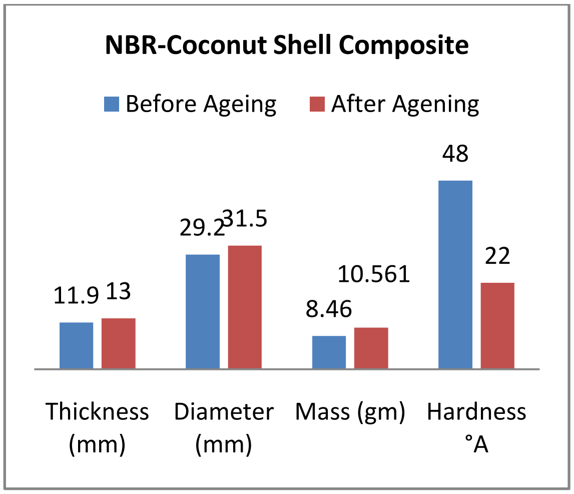

Fig : 4 Coconut shell- NBR composite

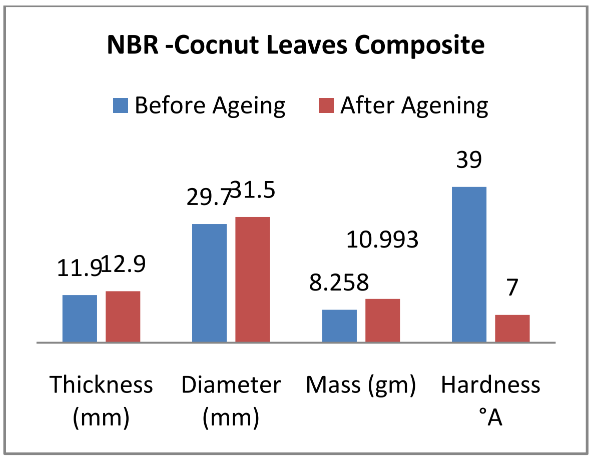

Fig:5 Cocnut leaves - NBR composite

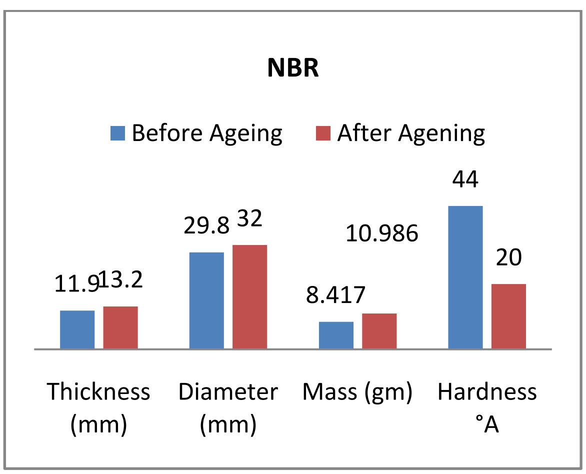

Fig : 6 NBR composite

Thus material was found compatible with commercial carbon based composite. The fuel pipe or hose pipe application was aimed and the composite with commercial matrix were prepared. It is observed from the Fig: 7 AS-NBR and CS-NBR are suitable for use of fuel tube and hose pipe in automobile industry.

Table 4 Result of petrol swelling test hardness, tensile test for NBR, Areca nut Shell, Coconut Shell

Sr. No	Sample	% Deviation in density	% Deviation in hardness	Remark
01	NBR	87.18	54.16	This is matrix material
02	AS-NBR	11	82	This can be used for fuel tube application
03	CS-NBR	28.3	54	This can be used for hose pipe application
04	CL-NBR	61.9	68	Needs to be studied further

3.4 Mechanical Properties of composite

In order to find out the processing condition the blends were tested rheological. Following are the results of the studies.

Table 5 Rheological behaviour of the samples

Sr. No.	Material	TS 1	TS 2	TS 3
01	Coco nut shell	1.21	1.39	1.93
02	Coco nut leaves	0.35	0.41	0.51
03	Areca nut shell	1.07	1.22	1.52

Activated carbon give rise to reinforcing effect and therefore the aggregate is smallest form of a given activated carbon grade well dispersed in an elastomeric that will still keep all the reinforcing capabilities of a filler. Activated carbon aggregates contain internal voids which are capable of absorbing polymer.

More specifically NBR rubber composite filled by activated carbon from Coconut shell and Areca nut shell showed better tensile strength as comparable with tensile strength of composite prepared from commercial carbon and NBR rubber.

For mechanical properties of rubber composites prepared from Coconut shell and areca nut shell activated carbon found improved at lower loading ratio of 20phr. Coconut leaves found very low values of mechanical properties as compared to the Coconut shell and Areca nut shell.

When it was tried to prepare rubber composite using 50phr formulation with commercial blend Elastorene 673 EL (70/30 NBR: PVC, W/W) the material scorched. This is well in

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relation with the results reported earlier by Geyuo et al [13] and Demrihan E. et al [14]. Mechanical properties decrease drastically after 30phr loading except that modulus can increase significantly against at 60 phr loading {Geyuo et al, 2008} but when the loading level is reached to limit value the activated carbon does not act as reinforcing filler {Demrihan E et al., 2007}.

Following are the photographs of the slab and button prepared for testing purpose.

Fig :7 Active Carbon -NBR buttons

Fig No: 8 NBR-PVC Activated Carbon button

Now it is under study to develop the carbon based commercial blend rubber composite for the fuel tube application.

IV. CONCLUSION

From the earlier studies and the results so far, we can conclude:

- Lignocellulose agricultural waste can be a starting material for the preparation of activated carbon.
- Elemental analysis and the BET study have shown surface modification of carbon.
- The coconut shell carbon and areca nut shell carbon were found to be competitive with commercial carbon in formation of rubber based composites for automobile applications.
- Looking at the tensile strength and hardness shown by the raw rubber based composite it is concluded that, acid activation of carbon improves reinforcement properties of the carbon
- There is certain scope to study the relation between chemistry of carbon activation and improved reinforcing ability of the carbon.

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