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FABRICATION AND CHARACTERIZATION OF FLY-ASH REINFORCED NATURAL RUBBER COMPOSITE

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Abstract— Rubber is a material which is used in many applications in automobiles, industrial and domestic. In general rubber is soft and high elastic material which can be derived from natural source. In this connection strengthening of rubber is very much essential when they are serving in high level of engineering applications for that unique types of filler materials are added with rubber matrix to improve some noble properties like wear resistance and thermal stability, Surface hardness and damping ability etc. To improve these properties we have chosen the humpty amount of available solid waste material in the world called fly ash from thermal power plants which consist of all poor heat conductive materials and high wear resistive abrasives like Al2o3, Sio2, Cao, Mgo. The fillers are added with the rubber matrix by diffusion bonding method which could get completed by two roll mill process. By choosing this surplus material as filler in rubber we can modify the properties and also reduce the consumption of other metallic fillers by which we can convert the industrial waste (fly ash) as an industrial wealth hence it is a kind of solid waste disposing technique. To compare the real significant we have prepared the samples by carbon and graphite also with same volume percentage what we have selected for Fly-ash. To check the effectiveness of addition Thermal stability, Wear, Hardness, Density, Scanning electron microscopy studies and spectroscopy analysis have done and evaluated the significant changes for each and every volume addition of filler. The forecasted areas of application of this material are auto mobile tyre manufacturing industries, Road construction, and hot fluid flow setups.

Keywords—Two Roll Mill, Pin on Disc, Scanning Electron Microscopy.

I. INTRODUCTION

A. COMPOSITE MATERIALS

It was found that when a lignite flyash is added to natural rubber composite the tensile strength, elongation at break, tear resistance and abrasion resistance decreases but hardness increases. Thus our main aim is to find out the effect of fly ash, when it is added to the natural rubber composite in different compositions to develop the hard material.

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II. MATERIALS

A. RUBBER
Rubber is an elastic material consist of polymer and water molecules in it and used for various application since it gives high tensile strength

1.1. NATURAL RUBBER

Natural rubber, also called India rubber or caoutchouc, as initially produced, consists of polymers of the organic compound isoprene, with minor impurities of other organic compounds plus water. Forms of polyisoprene that are used as natural rubbers are classified as elastomers. Currently, rubber is harvested mainly in the form of the latex from certain trees. The latex is a sticky, milky colloid drawn off by making incisions into the bark and collecting the fluid in vessels in a process called "tapping". The latex then is refined into rubber ready for commercial processing.

B.PULVERIZED FUEL ASH OR FLY ASH

Fly ash, also known as flue-ash, is one of the residues generated in combustion, and comprises the fine particles that rise with the flue gases. In an industrial context, fly ash usually refers to ash produced during combustion of coal. Fly ash is generally captured by electrostatic precipitators or other particle filtration equipment before the flue gases reach the chimneys of coal-fired power plants, and together with bottom ash removed from the bottom of the furnace is in this case jointly known as coal ash. We estimated fly ash class by EDTA method.

1.2 CLASS C FLY ASH:

Fly ash produced from the burning of younger lignite or subbituminous coal, in addition to having pozzolanic properties, also has some self-cementing properties. In the presence of water, Class C fly ash will harden and gain strength over time. Class C fly ash generally contains more than 20% lime (CaO). Unlike Class F, self-cementing Class C fly ash does not require an activator. Alkali and sulfate (SO4) contents are generally higher in Class C fly ashes.

C.CARBON

Carbon powder is a versatile powder that can be made from an allotrope of carbon, with each allotrope having a different use or function. Carbon powder, regardless of its allotrope, is typically used by metallurgists to make steel or to harden steel and iron.

D. GRAPHITE

Graphite is made almost entirely of carbon atoms, and as with diamond, is a semimetal native element mineral, and an allotrope. Graphite is the most stable form of carbon under standard conditions. Therefore, it is used in thermochemistry as the standard state for defining the heat of formation of carbon compounds. Graphite may be considered the highest grade of coal, just above anthracite and alternatively called meta-anthracite, although it is not normally used as fuel because it is difficult to ignite.

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III. FABRICATION

A.RUBBER SHEET MAKING

The rubber sheets added with fly ash particles can be manufactured by two roll mill process. Two roll mill is nothing but, a machine that uses shear force created by two horizontally positioned rolls rotating in opposite directions and different speeds relative to each other, in order to mix, refine, disperse, or homogenize viscous materials fed into it. Refer fig.1

Fig.1 Two Roll Mill

B.CURING PROCESS

Grain the natural rubber in two roll mill with filler and add accelerators i.e., MBTS (Dibenzothiazole disulphide) -10g,TMT (Tetra methyl thiruram disulfide) -5g, Sulfur -20g, Zinc oxide -25g added to it separately. This process is called Vulcanization. These additives modify the polymer by forming cross-links (bridges) between individual polymer chains. Vulcanized materials are less sticky and have superior mechanical properties. Refer fig .2

Fig.2 Curing Die with Hot plate

After curing process the rubber sheets are mixed with the chemicals like fly ash, carbon and graphite in required quantities. A total of seven specimens are prepared. This process is carried in a two-roll miller

Specimen 1: Natural Rubber
Specimen 2: Natural Rubber mixed with 15% of Fly Ash
Specimen 3: Natural Rubber mixed with 20% of Fly Ash
Specimen 4: Natural Rubber mixed with 15% of Carbon
Specimen 5: Natural Rubber mixed with 20% of Carbon
Specimen 6: Natural Rubber mixed with 15% of Graphite
Specimen 7: Natural Rubber mixed with 15% of Graphite

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Then these specimens are tested in laboratory to find their physical and chemical properties.

IV.TESTING

A.SHORE HARDNESS

The hardness of plastics is most commonly measured by the Shore® (Durometer) test.Both methods measure the resistance of plastics toward indentation and provide an empirical hardness value that doesn't necessarily correlate well to other properties or fundamental characteristics. Shore Hardness, using either the Shore A or Shore D scale, is the preferred method for rubbers/elastomers and is also commonly used for 'softer' plastics such as polyolefins, fluoropolymers, and vinyls. The Shore A scale is used for 'softer' rubbers while the Shore D scale is used for 'harder' ones. Many other Shore hardness scales, such as Shore O and Shore H hardness, exist but are only rarely encountered by most people in the plastics industry. The Shore hardness is measured with an apparatus known as a Durometer and consequently is also known as 'Durometer hardness'. The hardness value is determined by the penetration of the Durometer indenter foot into the sample. Because of the resilience of rubbers and plastics, the indentation reading my change over time - so the indentation time is sometimes reported along with the hardness number. The ASTM test method designation is ASTM D2240.The results obtained from this test are a useful measure of relative resistance to indentation of various grades of polymers. However, the Shore Durometer hardness test does not serve well as a predictor of other properties such as strength or resistance to scratches, abrasion, or wear, and should not be used alone for product design specifications. Shore hardness is often used as a proxy for flexibility (flexural modulus) for the specification of elastomers. The correlation between Shore hardness and flexibility holds for similar materials, especially within a series of grades from the same product line, but this is an empirical and not a fundamental relationship.

B.WEAR TEST

We conducted wear test by using a Pin-on-disk (CSM instruments).It is a laboratory test procedure for determining the wear of materials during sliding using a pin on-disk apparatus. The materials are tested in pairs under nominally non-abrasive conditions. The coefficient of friction can also be determined in this method. The pin/ball is mounted on a stiff lever, designed as a frictionless force transducer. The friction coefficient is determined during the test by measuring the deflection of the elastic arm.Wear coefficients for the pin and disk materials are calculated from the volume of material lost during the test. The control of the test parameters such as speed, frequency, contact pressure, time and environmental parameters (temperature, humidity and lubricant) allows simulation of the real life conditions of a practical wear situation. Controlled atmospheres of varying humidity or composition can be used.

C. TEAR TEST

Tear strength is defined as the force per unit thickness required to cause a nick out in a rubber when it is stretched, under constant rate, in a direction substantially perpendicular to the plane of the cut. The tear test can be performed using the tensile testing machine itself. There are different types of test pieces used for conducting the tear tests. Since the tear strength is susceptible of the nick cut, tests performed using

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the test pieces with a right angle nick gives better reproducibility of test results. Tear tests give an indication of the behavior of the vulcanized in tear initiation and tear propagation. We test conducted by IS3400:PART12. We cut the material into one of the tear shapes shown in the figure3. Load the specimen into tensile grips. Begin the test by separating the tensile grips at a speed of 20 inches per minute. End the test after sample break (rupture).

Fig 3. Tear test specimen

D.VOLUME SWELL TEST

The absorption of liquids by rubber is a diffusion-controlled process, and the effect of the liquid therefore depends on both the time of immersion and the thickness of the rubber. With adequate exposure time to a liquid, rubber swells to an equilibrium. The amount of swelling is used as a measure of resistance of a rubber to liquid, such as oil. Oil-resistant rubbers, of course, do not swell significantly in oil but they may swell substantially in other liquids. Nitrile-butadiene rubber (NBR) is an example of an oil-resistant rubber that finds wide use in seals and other components that call for this property. Volume swell testing (also known as Fluid Resistance, Change in Volume, Liquids Resistance) measures the deterioration of rubber and rubber-like compositions as it relates to the change in volume of the liquid that it is exposed to. It is critical to use an exact match of the liquid that will come into contact with the vulcanized in service to predict actual results. In this type of test the rubber is immersed in water/oil for 24 hours and then the effect of the liquid on rubber is tested by finding the change in volume of the material.

D.SCANNING ELECTRON MICROSCOPY:

A scanning electron microscope (SEM) is a type of electron microscope that produces images of a sample by scanning it with a focused beam of electrons. The electron beam is generally scanned in a raster scan pattern, and the beam's position is combined with the detected signal to produce an image. SEM can achieve resolution better than 1 nanometer. Specimens can be observed in high vacuum, in low vacuum, and (in environmental SEM) in wet conditions. The most common mode of detection is by secondary electrons emitted by atoms excited by the electron beam. The number of secondary electrons is a function of the angle between the surface and the beam. On a flat surface, the plume of secondary electrons is mostly contained by the sample, but on a tilted surface, the plume is partially exposed and more electrons are emitted. By scanning the sample and detecting the secondary electrons, an image displaying the tilt of the surface is created

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1. MICRO STRUCTURE:

The microstructure of fly-ash rubber composites were studied for the physical bonding with the cured natural rubber. The micro structure images were taken by Scanning electron microscopy where the particle images and matrix form have shown. The images were taken by the scanning electron microscopy having 1X106times of magnification. The maximum size we can achieve is 1µm levels. The Fly-ash particles have exposed the image such bonding of particle with rubber matrix. The Figure shows the composite where the filler is bounded with rubber matrix as a dispersion strengthening concept the load from the rubber to filler in the rubber matrix have zoomed much to see the resin bit .

V.RESULT AND DISCUSSION

A.SHORE HARDNESS:

We have conducted shore 'A' hardness for our specimens. We got different hardness value .due to this result we find our fly ash specimen must give equal hardness like carbon, graphite. Refer TABLE I

S.NO	SPECIMENS	HARDNESS
1.	natural rubber	42-45
2.	15% of Fly Ash	54-57
3.	15% Of carbon	47-50
4.	15% of graphite	51-53
5.	20% of flyash	49-52
6.	20% of carbon	59-62
7.	20% of graphite	51-53

B.WEAR TEST

We conducted wear test by using pin on desk apparatus. We use cylindrical specimen for pin on desk. Refer fig .4

Fig .4

Using below values as input for apparatus

Load = 4.903 N ~ 5 N
Sliding speed = 0.5 m/s
Sliding distance = 150 m

We calculated density of various fillers they are

Density of rubber - 0.92g/cm3
Density of fly ash - 2.17 g/cm3
Density of carbon - 2.26 g/cm3
Density of graphite - 2.23 g/cm3

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5.1 .Volume x density = weight

TABLE 2

Specimen	**Density g/cm³**	**Difference in gram**	**Difference of volume in cm³**	**Difference of volume in mm³**
NR	0.92	0.02	0.0217	2.17
20% of fly ash	3.09	0.1	0.0323	32.3
20% of carbon	3.18	0.08	0.0252	25.2
20% of graphite	3.15	0.78	0.2476	247.6

5.2. Wear rate = volume loss in mm3 / sliding distance in m

TABLE 3

Specimen	**Wear rate in mm³/m**
Natural rubber	0.1446
20% of flyash mixing with natural rubber	0.2153
20% of carbon mixing with natural rubber	0.168
20% of graphite mixing with natural rubber	1.650

C. TEAR TEST

We conducted tear test for the entire sample but tear is minimum for flyash composite because it is ceramic so did not get compatibility with the rubber matrix and also the particles are already an oxides so they could not react further but in carbon since it is a virgin base and have more valence it can easily react with the oxygen and forms corbon-di-oxide and carbon monoxide perhaps and create very good compatibility with rubber matrix. Refer table 4

TABLE 4

S.NO	SPECIMENS	TEAR STRENGTH in kg/mm
1.	natural rubber	2.80
2.	15% of Fly Ash	0.50
3.	15% Of carbon	3.08
4.	15% of graphite	1.11
5.	20% of flyash	2.06
6.	20% of carbon	3.11
7.	20% of graphite	1.90

D. VOLUME SWELL TEST

In this type of test the rubber is immersed in water/oil for 24 hours and then the effect of the liquid on rubber is tested by finding the change in volume of the material. Volume should be increased for oil more than water .Refer table 5

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TABLE 5

S.no	Time in hour	Liquid	Change in volume in %
1	24	ASTM OIL3	7.37%
2	20	WATER	0.75%

E.MORPHOLOGY:

1. Micro structure: The microstructure of fly-ash rubber composites were studied for the physical bonding with the cured natural rubber. The micro structure images were taken by Scanning electron microscopy where the particle images and matrix form have shown. The images were taken by the scanning electron microscopy having 1X106times of magnification. The maximum size we can achieve is 1µm levels. The Fly-ash particles have exposed the image such bonding of particle with rubber matrix. The Figure shows the composite where the filler is bounded with rubber matrix as a dispersion strengthening concept the load from the rubber to filler in the rubber matrix have zoomed much to see the resin bit .image are attached for reference. We conducted test for 20 % fly ash mix with natural rubber sample only in this particles are clear visible. Refer fig .9.

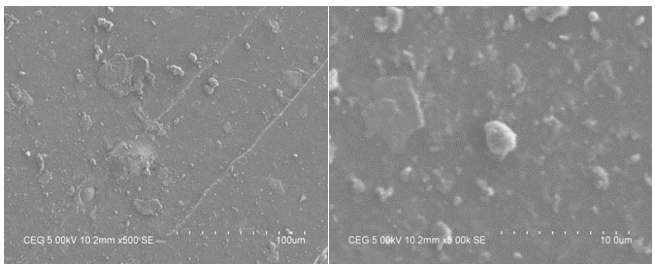

Fig .9 SEM Images of Rubber with 20% Flyash

VI. CONCLUSION

As per the test and the outcomes the addition of flyash industrial waste could modify the mechanical and thermal and also the chemical properties of natural rubber. The comparison has been taken with the traditional fillers like carbon and graphite we have checked the effectiveness of the significant addition of flyash material. So the waste disposal also could be maximized with the minimum level of environmental errors. In other side density of flyash loaded natural rubber materials are very less hence the weight versus volume ratio also reduced consistently it will leads the product manufacturing with less weight. Since the flyash is a bundle of ceramics they can serve the better job in mechanical and thermal properties and also it is noticed that the ceramics have wonderful chemical resistance and oil resistance properties that also has proven in the test. In the tear side though the decremented have absorbed and the greatness can be seen in the hardness and wear properties. The SEM results have shown the uniform dispersion of fly ash in the rubber matrix that also could serve the best. Thus the addition of flyash will make the significance in the natural rubber and it can be a best and cheapest filler instead of the traditional materials. At the same time the solid waste disposable also slowly happens by

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consuming the waste for industrial wealth thus the approach is a noteworthy process.

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