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# Fabrication And Chareactreization Of Particulate Carbon Composites

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Short Profile

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**ABSTRACT:**
Particulate composite consist of particles immersed in matrices such as alloys and ceramics. They are usually isotropic because the particles are added randomly. Particulate composites have advantages such as improved strength, increased operating temperature, oxidation resistance, etc. Composite samples were prepared to evaluate the effect on various properties of the samples heat treated at 1000oC. Optical microscopy was used to study the distribution of black, fly ash and coke particles in the carbon matrix and to observe other parameters. It shows that the reinforcing particles are well distributed in the carbon matrix and also exhibited good bonding between the matrix and reinforcing particles. Mechanical structural and physical properties of the composites were obtained.

**Key words:** Particulate, composites, carbon.

## INTRODUCTION :

Ceramic materials possess high strength and modulus at elevated temperatures. But their use as structural components is severely limited because of their brittleness. Ceramic materials are distinguished by a complex of properties previously unattainable with other groups of materials and they have a number of advantages over metallic materials: they have the capability to retain their properties in an oxidative medium at temperatures above 1200°C, good durability, excellent corrosion properties, low density and low thermal expansion, which makes them the only available materials for use in heat-loaded units and the components of prospective articles operating under oxidative conditions at high temperatures[1].

To develop a good understanding of composite behavior, one should have a good knowledge of the roles of reinfircement and matrix materials in a composite. The main functions of the fibers in a composite are to carry the load. In a structural composite, 70 to 90% of the load is carried by fibers. Fibers provide stiffness, strength, thermal stability and other structural properties in the composites [2-3]. They also provide electrical conductivity or insulation, depending on the type of fiber used. A matrix material fulfills several functions in a composite structure, most of which are vital to the satisfactory performance of the structure [4]. The important function of a matrix is to bind the fibers together and transfers the load to the fibers. It provides rigidity and shape to the structure. The matrix isolates the fibers so that individual fibers can act separately. This stops or slows the propagation of a crack. The matrix provides a good surface finish quality and aids in the production of net-shape or near-net-shape parts. The matrix also provides protection to reinforcing fibers against chemical attack and mechanical damage (wear). Depending on the matrix material selected, performance characteristics such as ductility, impact strength, etc. are also influenced [5-6]. A ductile matrix will increase the toughness of the structure. Literature reveals that most of the works reported on carbon and phenolic resin-carbon based composites were focused on processing and properties. Very little work seems to have been reported which describes the study of microstructure and effect on mechanical, thermal, and nitrogen adsorption properties of carbon and carbon-carbon composites. Most of the researchers have developed phenolic-carbon based composites involving the incorporation of carbon fillers. The present research work was undertaken to develop phenolic-carbon based composites using mixing and change in composition and processing parameters and to study their microstrutural, electrical and mechanical properties [7].

The present study was to develop carbon-composite using phenolic resin as a matrix system and carbon fillers as reinforcement agent. Sic as reinforcements are of specific interest since the combination of the materials, i.e. carbon and ceramic can produce specific characteristics difficult to obtain by any of the original components separately. SiC/carbon reinforced carbon particulate composites promise to retain good properties of both carbon and ceramic material, such as high mechanical strength, good resistance for high-temperature, corrosion, oxidation, thermal shock as well as electrical and thermal conductivity.

## 2.Raw materials and fabrication of composites

### 2.1 SiC/carbon reinforced particulate composites

### (a) Phenolic resin as matrix

The matrix precursor used in this research work was phenolic resin (Novolac type) mixed with 12 % hardener manufactured by Polymers and Polyols Chemicals, Vapi. The novolac phenolic was used as precursor for composite preparation due to its good dimensional stability and high yield. Prime function of phenolic resin was to bind or hold all the ingredients [8]. Phenolic resin is a common binder for resin-based carbon materials. Thermal applications of pure phenolic resins derived carbons are usually limited due to their relatively poor strength. Therefore, it is imperative to incorporate various reinforcing and filling constituents such as reinforcing fibers and fillers into phenolic resin-based composites with the purpose of increasing their strength and stability [9-10].

During carbonization stage, non-carbon elements in the cured resin come out as volatiles, such as $H_2O$, $CO$, $CO_2$, $H_2$ and other gases. The reactions were due to the condensation of aromatic ribbon molecules in the cured resin and the volatilization of low molecular weight species. This results in shrinkage and weight loss in the cured resin. The carbon yield of the phenolic resin was found to be (~ 56.74%) which shows it is a good precursor for the carbon-ceramic products.

### (b) Carbon black as filler

The conductive filler HAF-330 carbon black in the powder form (particle size 2-12 μm) from waste rubber tire was procured from Graphite India Limited. Bangalore and was used for studies in this work. Carbon black particles have the graphite-type crystalline structure, providing excellent electric conductivity [12]. Therefore, carbon black is widely used as conductive filler, being mixed in plastics, elastomer, paints, adhesives, films, and pastes. Carbon black is also used as an excellent antistatic agent [13]. Carbon black also provides stable resistance, and therefore is used as electronic equipment related material in various display components, magnetic recording materials, and OA rolls [14].

### (c) Fly ash as filler material

Fly ash was obtained from one of the thermal power station in Gujarat. Fly ash is composed of fine size particles (mean size 10–30 μm) of ceramic. Fly ash particles are typically generated at very high temperatures, i.e. above 1000oC. Hence, these are thermally stable for high-temperature applications and can be used as fillers in carbon particulate composites. Also, majority of fly ash contains substantial amount of silica, alumina, calcium sulfate and unburnt carbon in them, which are already being used in many of the composites fabrication. Fly ash is also readily available at very low cost [15]. Fly ash is a pozzolanic material and has been classified into two classes, Class F and Class C, based on the chemical composition of the fly ash. According to ASTM C 618, the chemical requirements to classify any fly ash are shown in **Table 2.1** [16].

| Properties | Class F | Class C |
|---|---|---|
| Silicon dioxide ($SiO_2$) plus aluminum oxide ($Al_2O_3$) plus iron oxide ($Fe_2O_3$),min,% | 70.0 | 50.0 |
| Sulfur trioxide ($SO_3$), max, % | 5.0 | 5.0 |
| Moisture Content, max, % | 3.0 | 3.0 |
| Loss on ignition, max,% | 6.0 | 6.0 |

Table 2.1: Chemical composition of two different classes of fly ash.

### (d) Calcined petroleum coke (CPC)

Calcined petroleum coke (CPC) was procured from Schutz Carbon Electrode Private Limited, Kalol, Gujarat. Calcined petroleum coke (CPC), which mainly comprised of graphitic carbons can be used as for carbon composite products for friction applications where low coefficient of friction is required [18]. During carbonization, these also add to the carbon yield. Petroleum coke (often abbreviated Pet coke or pet coke) is a carbonaceous solid derived from oil refinery coker units or other cracking processes. Calcined petroleum coke (CPC) is the product from calcining petroleum coke [19, 20]. The green coke must have sufficiently low metals content in order to be used as anode material.

In this work carbon black, fly ash, coke and combination of these fillers (reinforcements) with phenolic resin were used to make composites. In this work, compositions of carbon black, fly ash and coke are as per given in **Table 2.1.**

| ID | Novalac Phenolic resin | Carbon Black | Fly ash | Coke |
|---|---|---|---|---|
| 3R7CB | 30% | 70% | -- | -- |
| 3R4CB3F | 30% | 40% | 30% | -- |
| 3R2CB3F2C | 30% | 20% | 30% | 20% |

Table 2.1: Composition for carbon- particulate composites.

## 3. Result and Discussion

### 3.1 Physical properties of carbon-particulate composites

Carbonization involves pyrolysis of the thermosetting matrix which resulted in change in weight, density and exhibited dimensional shrinkage. If not controlled properly, pyrolysis may also leave behind pores and voids in the C-particulate composites. All these factors affect the density of CFC composites as well as their microstructure. Porosity in the C-particulate composites affects the structural stability.

### 3.1.1 Weight loss and volume shrinkage of C-particulate composites during carbonization

Heat treatment of the composites up to 1000oC involves pyrolysis of phenolic resin matrix. Weight loss of the composites is primarily due to the phenolic resin since the reinforcements viz. Carbon black, fly ash, and coke are already calcined materials and have been processed at temperature beyond 1000oC. The **Fig. 3.1 (a)** and **(b)** shows the variation in percentage of weight loss and volume shrinkage of composites with the same amount of phenolic resin in the composites. The addition of different kinds of reinforcing fillers with various concentrations, the weight loss and volume shrinkage increases. It has been found that addition of coke (3R2CB3F2C) didn't change the weight loss and volume shrinkage much compare to addition of fly ash (3R4CB3F). After this, the slope changes which may due to presence of more amount of fillers. Encapsulation of resin mass within filler might be changing the release of pyrolysis products and which get repyrolysed and hence change the ultimate weight loss.

At 1000oC, most of the oxides present in the composites undergo chemical reaction with carbon matrix (carbothermal reduction) to form porous carbide composites. These plots reveals that composites containing higher percentage of fly ash exhibit higher weight loss due to carbothermal reduction. As the weight loss is higher than the volume shrinkage percentage, these composites exhibit greater porosity.

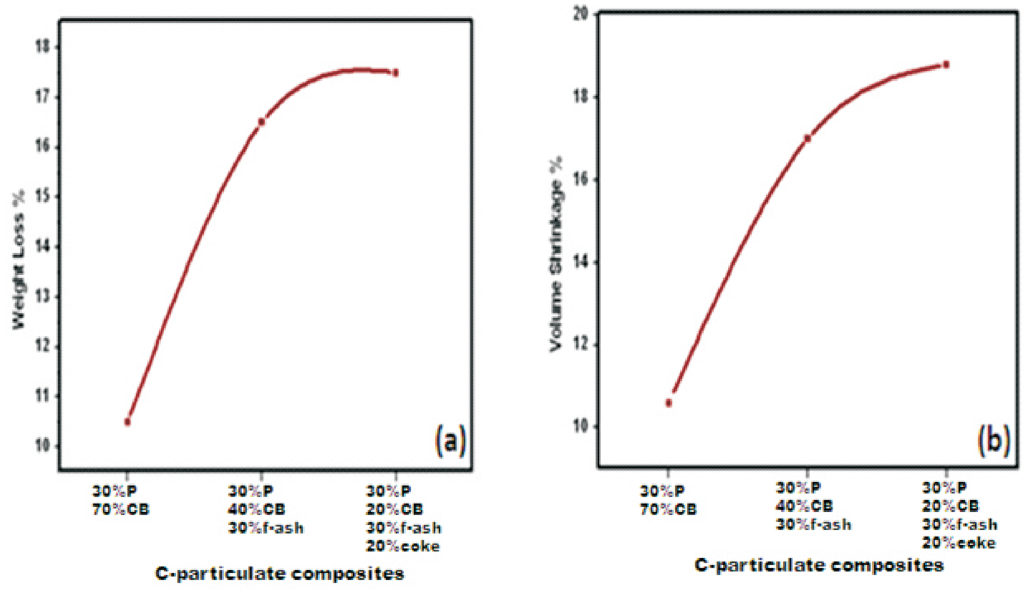

**Fig 3.1: (a) Weight loss and (b) Volume shrinkage during carbonization of C-particulate composites made with various reinforcing fillers.**

### 3.2.2 Density and porosity of C-particulate composites

For group –II these properties are plotted in **Fig 3.2 (a)** and **(b)**. It is clearly seen from **Fig.3.2(a)** that the density of C-particulate composites decreases with the heat treatment temperature. The densities of green C-particulate composites were measured as 1.54 g/cm3, 1.23 g/cm3 and 1.31 g/cm3 for 3R7CB, 3R4CB3F and 3R2CB3F2C respectively. In case of carbonized C-C composites it is clear that the density is decreased with increase in temperature. This is because of the shrinkage in matrix (phenolic based carbon) during carbonization (heating) and due to reinforcements (carbon black, Fly ash and coke) gets dispersed in the carbon matrix so as to decrease the density at 1000°C.

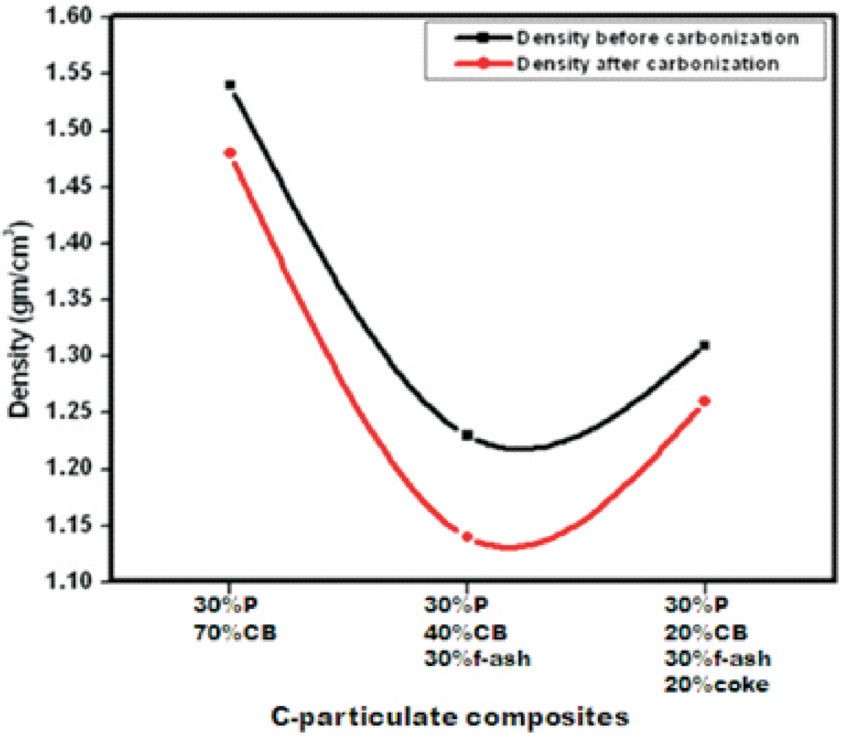

Fig 3.2 (a): Variation in densities of C-particulate composites (II) heat treated up to 1000°C.

With the addition of fillers in carbon black reinforced phenolic resin composites, like fly ash it is found that there is a reduction in density. There is a change in increase in density in case of 3R2CB3F2C composites which is due to presence of coke content in C-C composites. 3R7CB has 1.48 g/cm3 carbonized density and 3R2CB3F2C has about 1.25 g/cm3 carbonized density. 3R7CB has 70% of total carbon black content, 3R4CB3F has 40% carbon black with 30% fly ash and 3R2CB3F2C has 20% carbon black, 30% fly ash and 20% coke as reinforcement. In all three samples 30% phenolic resin as matrix is present. This implies that during carbonization the filler particles get dispersed in the carbon matrix leading to decrease in packing density [21]. Heat treatment also leads to change in density and porosity resulting from evolution of pyrolysis products.

With the addition of fillers like fly ash which is consists of oxides has density higher than carbon but density of carbon-fly ash composite decreases because composites having fly ash exhibit higher apparent porosity at all stages due to less compaction. The abundance of fly ash particles hinders compaction of the total mass and binding of the reinforcing particles. The inclusion of coke increases the density of the 3R4CB3F composites. There is an increase of 10-11 % density of 3R2CB3F2C composite over 3R4CB3F at 1000°C.With the addition of coke the density of the sample increases because the addition of filler particles get compact in carbon matrix leading to increase in packing density and reduction in porosity **[Fig.5.13 (b)]**. Therefore, 3R2CB3F2C has higher density as compared to 3R4CB3F. This is clearly seen from its optical micrographs **[Fig.5.14]**.

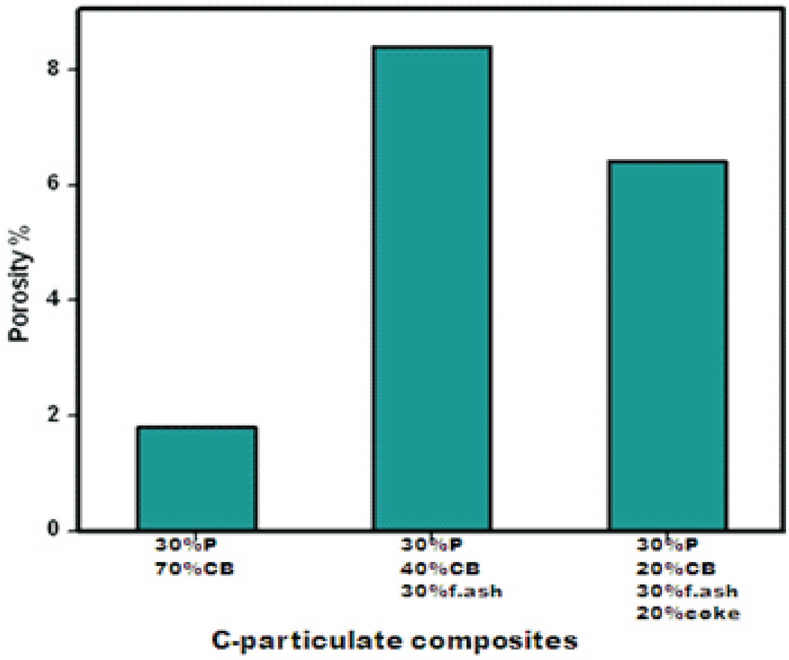

**Fig 3.2(b): Variation of % porosity in C-particulate composites heat treated to 1000oC.**

## 3.2 Macro / Microstructure studies

Microstructural investigation was done to see the microstructure as well as morphology of C-particulate composites heat treated up to 1000°C. Both matrix as well as reinforcing agents (carbon black powder, fly ash and coke) was observed under optical microscopy and SEM. Macrostructure of the composites, distribution of the micro particles, presence of voids and generation of pores were examined by optical microscopy. Scanning electron microscopy was used to investigate the reinforcement-matrix bonding, porosity and reinforcement distribution as well as overall morphology of the as such and carbonized composites.

### 3.2.1 Optical microscopy of carbonized C-particulate composites

The Optical micrographs illustrates the microstructures of C-particulate composites with 70 % weight fraction of carbon black powder (reinforcing filler) and with the addition of fly ash and coke with 30% phenolic resin at 50x magnification (Fig. 3.3). The purpose of microstructure examination was to investigate the particle size, shape morphology and distribution of the carbon black, fly ash and coke with their interfacial integrity between the matrix and reinforcement and the effect of reinforcing filler particles.

Optical microscopy was used to investigate the distribution of carbon black, Fly ash and coke particles in the carbon matrix. **Fig. 3.3** shows optical micrographs of the 3R7CB, 3R4CB3F and 3R2CB3F2C composites, heat treated at 1000oC. These images show that the reinforcing particles are well distributed in the carbon matrix (phenolic resin). These also exhibit good bonding between the matrix and reinforcing particles. In case of 3R7CB, voids are seen at the interface as well as within the matrix itself **[Fig.3.3 (a)]**. As seen in **Fig.3.3 (b)** and **3.3(c)**, fly ash and coke are also well distributed and well bonded with the carbon matrix. The matrix region as well as fly ash rich regions exhibit large amount of porosity (micro porosity, voids, etc.), which justified the nature of density and %porosity plots of these samples.**[Fig. 5.13 (a)and (b)].**

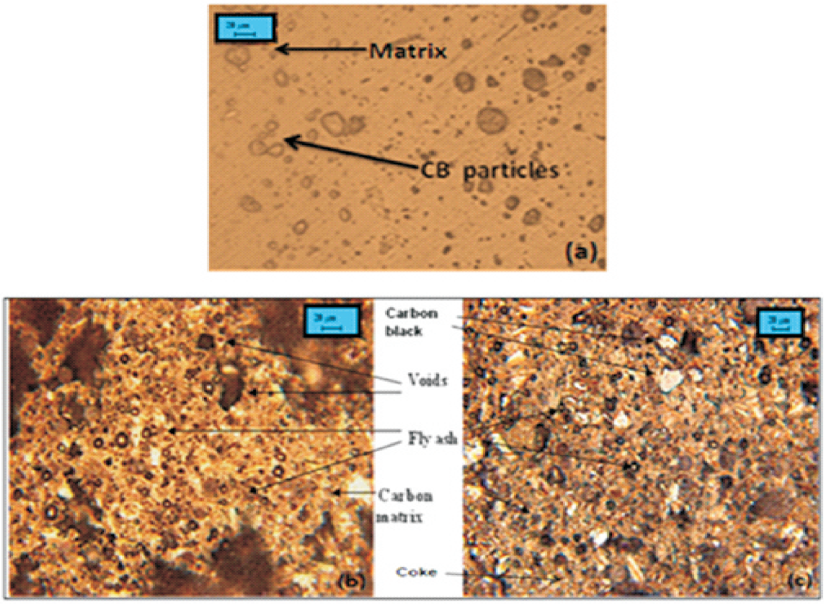

Fig 3.3: Optical micrographs of C-particulate (II) composites (a) 3R7CB. (b) 3R4CB3F (c) 3R2CB3F2C.

### 3.2.2 Scanning electron microscopy of carbonized C-particulate composites

SEM micrograph of 3R4CB3F **[Fig. 3.4 (b)]** composite shows that most of the particles are spherical in shape. Some of the particles are not spherical, which are irregular shaped carbon particles. These particles are seen in different forms, both anisotropic as well isotropic forms in the fly ash [22].

The microstructure consists of three main constituents. Fly ash particles are having spherical shape whereas carbon black particles showed irregular shape **(Fig: 3.4)**.The large and small dark regions describes the voids present in C-particulate composites. Remaining regions show presence of carbon matrix. SEM micrographs of **Fig.3.4** shows that addition of fly ash in 3R7CB composite, the inter particle distance increases that consequently reduces the density of composites. In 3R7CB **[Fig.3.4 (a)]** composite the porosity is less compare to 3R4CB3F and 3R2CB3F2C and it has maximum density as shown in the SEM images **(Fig.3.4).**

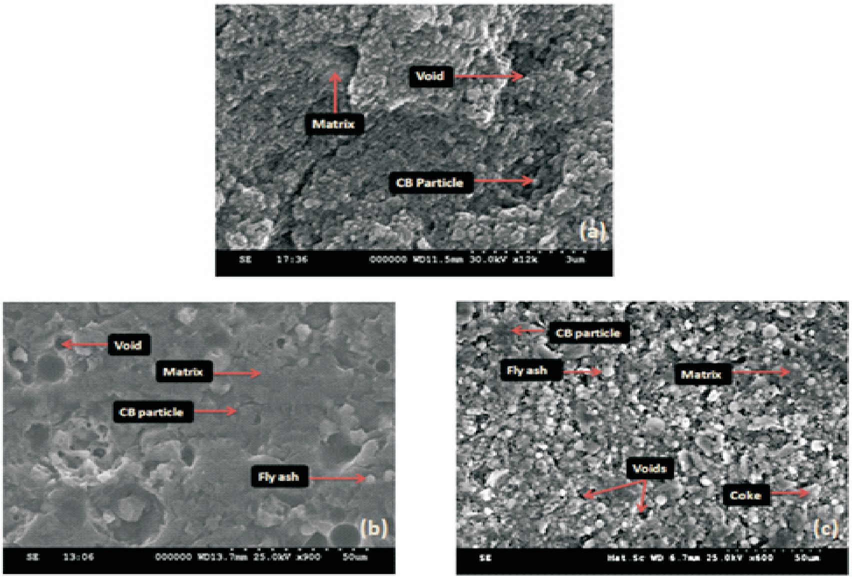

Fig 3.4: SEM micrographs of C-particulate composites (II) (a) 3R7CB (b) 3R4CB3F and (c) 3R2CB3F2C.

The **Fig.3.4 (a)** shows strong bonding between carbon black particles and matrix. It also indicates the dense structure of 3R7CB composite compared to other two composites. The **Fig.3.4(b)** shows SEM image of 3R4CB3F composite containing fly ash heat treated to 1000°C. The Fig.3.4 (b) shows voids in composite confirming from optical microscopy. It also indicates the weak bonding between fly ash particles and carbon matrix. Surface activation of fly ash particles occurs due to ball milling. New generated surfaces have less adhesive capabilities and thus resin particles get dispersed to the surface of the reinforcing particles. It also shows the distribution of fly ash and carbon black particles in the carbon matrix (3R7CB&3R4CB3F). This **Fig.3.4 (c)** also reveals the dense structure of the 3R2CB3F2C composite after carbonization. All the SEM images show that there is uniform distribution of reinforcements in the carbon matrix.

## 3.3 Mechanical properties

The mechanical properties of the 3R7CB, 3R4CB3F and 3R2CB3F2C composites generally depend on the amount of reinforcing materials, shape, porosity/cracks, interfacial bonding between reinforcement and matrix in the composites and the characteristics of other reinforcements.

Evaluation of mechanical properties for C-particulate composites was determined by means of Rockwell hardness and compressive measurement.

### 3.4.1 Rockwell hardness of carbonized C-particulate composites

The composites developed in the present studies consisted of phases of different hardness. Phenolic derived carbons are glassy carbons and hard whereas cokes are soft. Therefore, hardness of the resulting composites will depend on the amount and distribution of hard reinforcing materials as well as on the bonding between matrix and reinforcement and the factor like porosity [22]. The composites were analyzed for Rockwell hardness and results are presented in **Fig.3.5**. The hardness of 3R7CB sample is maximum compared to other two samples that are due to strong bonding between CB particles and matrix and also because of least porosity. This result is justified from the microstructural analysis **[Fig.3.3 (a)** and **Fig.3.4(a)]**. The composite do not exhibit any appreciable increase in hardness on addition of fly ash (3R4CB3F), rather it is found to decrease with addition of fly ash content. However, the decrease in hardness value is more. This is attributed to high porosity of the composites, which is also justified by optical image **Fig.3.5(b)**.The hardness of the carbonized 3R4CB3F composite is found to be lower than carbonized 3R2CB3F2C composites.

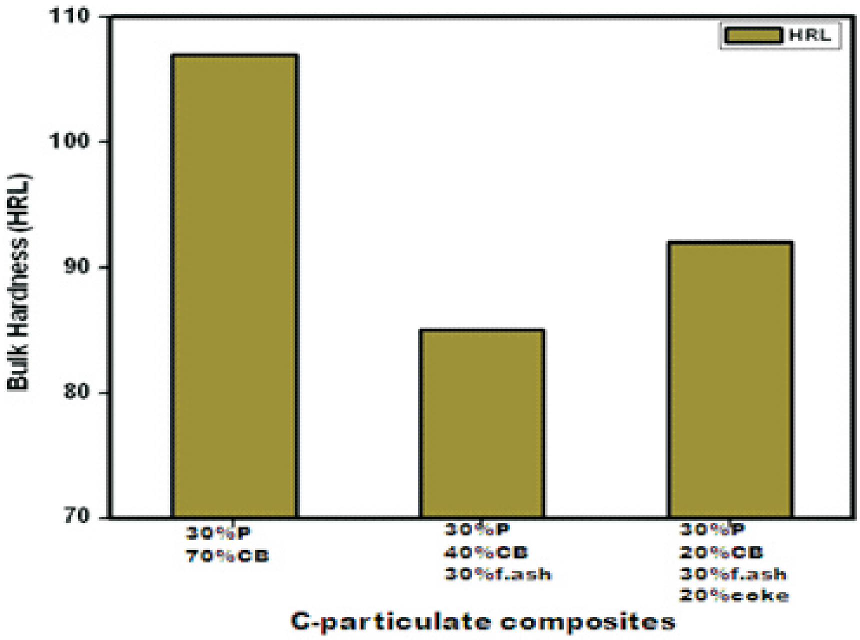

**Fig 3.5: Rockwell bulk hardness of C-particulate composites (II).**

Microstrutural investigation **[Fig.3.3 (C)** and **Fig.3.4 (C)]** clearly shows that the addition of coke (3R2CB3F2C) results in a better bonding among reinforcing fillers and matrix over 3R4CB3F. This reduces the porosity and void contents of composite [23].

### 3.4.2 Compressive strength measurement

The **Fig. 5.17** (Group II) shows the effect of different constituents present in the C-particulate composites on the compressive strength of the composites. The 3R7CB composite containing 70% carbon black has highest compressive strength value compare to other two composites.

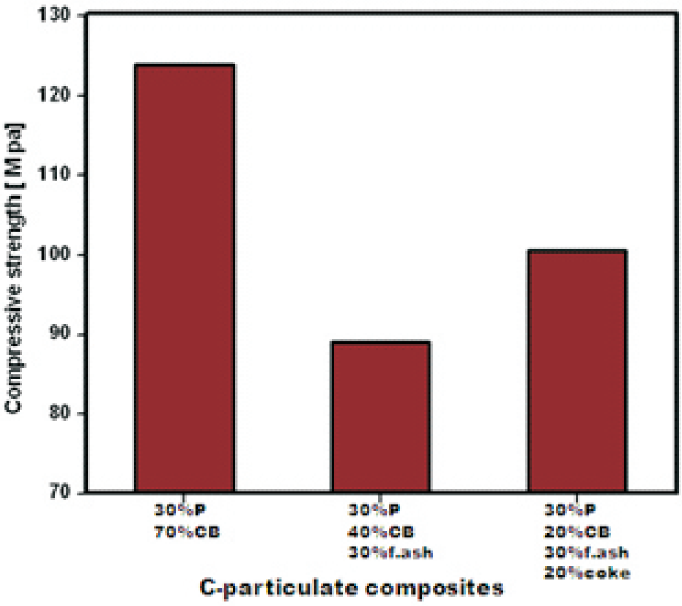

**Fig 3.6: Compressive strength of the C-particulate composites (II).**

This may be attributed to the strong bonding of carbon black particles with carbon matrix [24]. The strong bonding nature of carbon black particles with carbon matrix can be seen in micrographs which is shown in **[Fig 3.3 (a)** and **Fig. 3.4(a)]**. It is clearly seen from **Fig.3**.6that addition of fly ash decreases the compressive strength of the composite. Addition of only 30% fly ash with 40 % of carbon black drastically decreases the compressive strength of the 3R4CB3F composite. This reduction may be attributed to the weak bonding of fly ash particles with carbon matrix as compared to carbon black particles. This can be seen in micrographs shown in **[Fig.3.3 (b)** and **Fig 3.4 (b)]**. However, the addition of fly ash decreases the bonding between reinforcement and matrix and simultaneously increases the porosity. This decreases the compressive strength of the composite. The addition of coke (3R2CB3F2C) which is soft material makes the strong bonding between fillers and matrix, consequently increases the compressive strength of this composite compare to 3R4CB3F. The value of compressive strength is 100 MPa, which is higher as compared to 3R4CB3F composite. This indicates that the addition of coke leads to improvement in the compressive strength of the 3R2CB3F2C composite. So, improvement in the compressive strength by introducing the coke particles could be explained by the homogeneous distribution of the coke particles in carbon matrix **[Fig. 3.3(a)** and **Fig.3.4 (b)]**. Increase in homogeneity decreases the pores present in 3R2CB3F2C composites which finally increases the compressive strength.

## 3.5 Electrical properties (Bulk DC conductivity)

The electrical conductivity of the phenolic resin is about $3.05\times10^{-15}$ mho.cm$^{-1}$, that shows that it possesses the good electrical resistance or other way it is a good electrical insulator. The electrical properties altered with the addition of CB particles in to the phenolic resin (3R7CB). The composites behavior evolves from insulating material characteristics to those of conductive materials with the addition of conductive fillers. The effective utilization of carbon black in composite applications depends strongly on the ability to homogeneously disperse them throughout the matrix without destroying their integrity [25]. The conductivity of the composites increases with increasing CB content. It is due to the formation of the conductive network of CB particles through the polymer matrix and thus, composite becomes conductive. These conductive networks or paths have either physical contact between CB particles and/or they are separated by a very small distances across which electrons can tunnel. This shows the appearance of microstructure of CB filled phenolic resin composites. At 70 wt% concentration of CB particles conductivity increased drastically to 100 mho.cm-1. This may attributed to the because of hopping and tunneling of charge carriers through the matrix. The fly ash is very poor conductor of electricity so the addition of fly ash particles (3R4CB3F) in to the composite opposes the formation of conductive networks and thus suddenly reduces the bulk DC conductivity of the composites **(Fig.3.7)**. This reduction in bulk DC conductivity may be attributed to the weak bonding of fly ash particles with carbon matrix as compared to the carbon black particles. However, the reduction in electrical conductivity is high, as 30% of fly ash is added in to the composite. This is attributed to the high porosity of the composites due to the addition of fly ash. This is also justified by optical image **[Fig.3.3(b)]**.The addition of coke (3R2CB3F2C) improves the bulk DC conductivity of the composite which is due to the graphitic nature of coke [26]. Also micrographs observation **[Fig.3.3 (c)** and **Fig. 3.4 (c)]** clearly show that the addition of coke improves the bonding and reduces the porosity compare to 3R4CB3F composite.

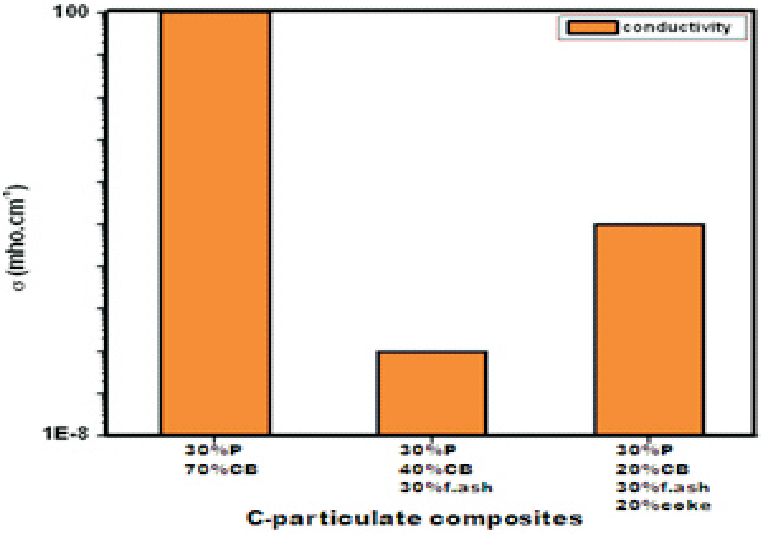

**Fig.3.7: Bulk DC conductivity test of carbonized C-particulate composites (II) (a) 3R7CB (b) 3R4CB3F, (C) 3R2CB3F2C samples.**

## 4. Conclusion

Characterization of first group shows that process of carbonization improves the density and reduces the porosity that justified by its macro/micro studies. Addition of graphite powder decreases the composite hardness because of weak bonding of Van der Walls forces between the layers.

Homogeneous distribution of SiC particles improves the hardness and compressive strength and simultaneously reduces the composite porosity. An electrical conductivity measurement shows that increasing the graphite powder enhances the conductivity but at the same time addition of SiC particles drastically reduces the electrical conductivity.

In second group 3R7CB composite possesses the maximum density that reduces after carbonization because of shrinkage of base matrix and particles dispersion increases the composite porosity which clearly seen in optical micrographs. Bulk hardness of 3R7CB is maximum compare to 3R4C3F and 3R2CB3F2C. The sample 3R7CB is more electrically conducting composite compare to other two composites because of its least porous structure and conducting filler.

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