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Comparative performance assessment of cenosphere and barium sulphate based friction composites

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ABSTRACT

Friction composites utilising cenosphere and barium sulphate as major fillers were fabricated both separately and in combination. Thermal analysis revealed initial degradation temperature >500 °C irrespective of the composition. Mechanical properties viz. hardness, compressibility and shear strength were found to be well above the standard values as per the industrial practice. Dynamic mechanical response demonstrated improved storage and loss moduli for barium sulphate based composites. Tribological assessment conforming to the regulation-90 as per the Economic Commission for Europe (ECE) norms revealed improved wear resistance, enhanced recovery, lower disc temperature rise and reduced friction fluctuation for cenospheres based composites. Composites containing both fillers showed cenospheres dominated tribological response. Empirical correlation between wear, friction coefficients and dynamic mechanical properties as temperature controlled parameters was developed to predict wear. Worn surface analysis of the brake pads was carried out by scanning electron microscope (SEM) to visually analyse the associated wear mechanism at the braking interface through various topographical attributes viz. primary and secondary contact plateaus.

1. Introduction

Increasing attention on the use of sustainable and environmental friendly resources, without depleting mineral resources has caused a paradigm shift in research and developmental activities towards providing solutions which meet the sustainability and techno-commercial viability objective. Friction materials such as brake pads, brake linings and brake shoes are comprised of four basic ingredients viz. binder (usually phenolic resin and its modified forms), fillers (barium sulphate, vermiculite, mica etc.), fibres (glass fibres, kevlar fibres, lapinus fibres etc.) and friction modifiers (abrasives and solid lubricants). Among these prime ingredients, fillers occupying large volume fractions in friction material formulation are important as they reduce cost and facilitate processing of the composites [1]. Designing friction materials meeting the sustainability objective requires replacement of rapidly depleting mineral resource based ingredients in friction material formulation with alternative resources which are sustainable and cheap. Conventionally, mineral fillers such as barium sulphate, calcium carbonate, mica etc. are widely used as functional fillers in friction materials for manufacturing automotive brake pads. On the other hand, flyash, a coal combustion by-product and its refined form i.e. cenospheres have emerged as potential fillers for brake applications [2–8] not only because of good mechanical and tribological properties obtained but also due to the fact that there is a value addition aspect associated to the otherwise costly and improper disposal of this industrial byproduct. A recent study pertaining to the comparative assessment of flyash and cenospheres filled composites has revealed enhanced wear resistance and improved recovery characteristics accompanied with optimum friction performance for cenospheres based brake composites [8]. Apart from benchmarking studies of propriety nature carried out in industries, there is a dearth of studies pertaining to comparative performance evaluation in open literature. Cenospheres with its many associated advantages such as low bulk density (0.26–0.45 g/cc), low thermal conductivity (0.11 W/mK), high hardness (5–7 Mohs scale) and hollow spherical morphology will provide a suitable and cheaper alternative to costlier fillers used in the industries [9,10]. Friction materials usually contain multiple and often functionally disparate ingredients and hence developing a formulation based on optimised tribological performance characteristics is a tough task faced by development engineers. Dadkar et al. [4], Jaggi et al. [5] and Satapathy et al. [6] utilised sequential dilution of the resin with the numerous ingredients one by one with flyash as major constituent

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and have been quite successful in reaching a formulation with optimised performance defining attributes (PDA's) such as friction-fade, friction-recovery, maximum disc temperature rise (DTR) and wear.

Performance of brake friction composites is dependent on both composition as well as operating parameters. There have been numerous studies [11–13] involving a change in nature/amount/type of one ingredient, keeping all other ingredients constant, to determine the influence of that particular constituent on the various performance defining attributes. Cho et al. [14] utilising OVAT (one variable at a time) ideology concluded that thermal stability, hardness and morphology of the constituents decide the fade and wear resistance of the composites. Rhee et al. [15,16] have carried out pioneering research towards correlating friction and wear with operating variables such as normal load, sliding speed and time and proposed empirical equations for their prediction. Friction and wear prediction a priori is difficult due to multi-component and heterogeneous nature of friction materials. Designs of experiments based techniques have gained wide usage in research as well as in industrial communities for determining the simultaneous effect of operating variables on the tribological performance [8,17]. Morphological characterisation post performance testing is of paramount importance for identifying the wear mechanisms involved during braking cycles as well as to unravel the composition specific variation of friction characteristics through various topographical features such as primary and secondary patches formed overtime during the testing. In this regard, Jacko et al. [18], Eriksson et al. [19] and Osterle et al. [20] have done extensive work to explain the mechanism of friction layer formation and its dependence to the friction and wear characteristics.

The purpose of the present work is thus to comparatively evaluate the performance of cenosphere filled composites with barium sulphate filled composites in terms of tribological, morphological and mechanical properties. It further deals with the development of an empirical equation for correlating wear with both tribological and dynamic mechanical properties.

2. Experimental

2.1. Materials and fabrication of composites

Cenospheres, having bulk density (0.26–0.45 g/cm^3), average particle size (55 μm) and loss on ignition (2% max), were obtained from Indian Industrial Exporters (IIEX), New Delhi, India. Scanning electron microscope (SEM) is used to characterise cenospheres for their morphology. Particle size and particle size distribution were assessed with the help of *Image J* software (National Institutes of Health, Bethesda, Maryland, USA). In the composition mix, apart from varying the cenospheres, barium sulphate and glass fibres content, the amount of phenolic resin (novolac type JA10), kevlar pulp (Twaron-IF258), graphite, lapinus fibres (Rockbrake® RB250-Roxul® 1000) and vermiculite were kept fixed. After weighing, the ingredients were mixed sequentially with powdery ingredients first followed by the fibrous ingredients for 20 min in a plough type shear mixer (cylindrical vessel of diameter 9.5″ and length 8.5″) facilitated with rotor (300 rpm) and chopper (3000 rpm) to ensure the homogeneous mixing. After mixing, preforming of the mix in a press was carried out followed by moulding of the preformed mix in a compression moulding machine having platen temperatures of 155 °C and pressure of 125 kg/cm^2 for 5 min with intermittent breathing steps to drive out any volatiles evolved during the curing. Brake friction composites were prepared by compression moulding of the composition mix as shown in Table 1. After moulding the composite specimens were post cured for 5 h at 165 °C to complete curing as well as relieving any residual stresses developed during the moulding cycle.

Table 1
Friction composite formulation and their designation.

Ingredients by wt%	Composite designation				
	CG0	**CG5**	**BG0**	**BG5**	**BCG5**
PF resin	15	15	15	15	15
Cenosphere	60	55	–	–	27.5
Barium sulphate	–	–	60	55	27.5
Glass fibre	0	5	0	5	5
[a]Fibrous ingredients	15	15	15	15	15
[b]Functional fillers	10	10	10	10	10

[a] Kevlar:lapinus = 1:2.
[b] Vermiculite:graphite = 1:1.

2.2. Physical and mechanical characterisation

The composites were characterised for their physical (density, porosity and ash content) and mechanical properties (hardness, shear strength and compressibility) and the results obtained are presented in Table 2 and further discussed in Section 3.2. Density was determined using Archimedes's principle by measurement of the weight of the specimen in air and in water as reported elsewhere [21].

Acetone extraction was carried out in a Soxhlet extraction apparatus for estimation of the amount uncured resin content. Porosity was determined following JIS D 4418:1996 standard specifications; it involves immersion of specimen in oil for 24 h (8 h heating at 90 °C and 16 h cooling at room temperature). Ash content was determined by gravimetric methods, which included heating the composites to approximately 850 °C in a muffle furnace. Hardness (defined as the resistance to local deformation) was measured by Rockwell hardness tester from Fine Testing Machine to confirm proper curing and uniform mixing of the composites. Shear strength, a measure of composite integrity and its adhesion with the back plate, was measured on Universal testing machine from Fuel Instruments & Engineers Pvt. Ltd. whereas compressibility, a measure of change in thickness under graded pressure, was measured on hydraulics compressibility testing machine from Hydro Plus, India.

2.3. Thermal characterisation

Thermal analysis of the neat phenolic resin and fabricated composites were carried out on differential Scanning calorimetry (DSC) and thermal gravimetric analysis (TGA) respectively. DSC was performed on a Q200 machine from TA Instruments USA, in an inert atmosphere (nitrogen) at a heating rate of 10 °C/min from room temperature to 350 °C, to analyse the curing characteristic of phenolic resin. TGA was carried out on a Pyris 6 TGA instrument, in an inert atmosphere (nitrogen) at a heating rate of 20 °C/min from room temperature to 850 °C, to determine the thermal stability of the fabricated composites.

2.4. Dynamic mechanical properties characterisation

Dynamic mechanical analysis of the test specimens, with dimensions of $38 \times 10.3 \times 3.3$ mm^3, have been performed on a Q800 machine (TA Instruments, USA) to characterise the storage modulus (E'), loss modulus (E'') and loss-tangent (tan δ). The test specimens were subjected to a sinusoidal displacement at constant strain amplitude with a frequency of 1 Hz in the temperature range of 35–350 °C and at a heating rate of 5 °C/min.