ABSTRACT

Textile-rubber composites are used in many applications like tire cord, conveyor belt, hose, power transmission belt, inflatable and non-inflated structures, hovercraft skirt, snowmobile track and such others. In textilerubber composites, as the application demands, polyamide fibres, nylon, has been used widely as reinforcements due to superior tensile elongation, elasticity at high elongation and strength. In the composite, the adhesion between the rubber and the reinforcement plays an important role in ensuring the serviceability of the composite under adverse conditions. The adhesion strength between the matrix and reinforcement is not just for holding the materials together, but also for providing capacity to withstand the operating load, transfer the load stress from the rubber matrix to the reinforcing fibre materials. Hence, the adhesion properties of reinforcements with the matrix are always preferred to be high.

Nylon, being the suitable candidate for rubber composite, is unable to adhere with rubber directly, leading to poor interfacial adhesion. Some of the factors that prevent the adhesion of rubber to the fibre surface are smooth polymer surface, hydrophobic and low reactivity characteristics of nylon, presence of spin finish and size ingredients applied during manufacture. In order to overcome this problem, at present, the fibres are first coated with adhesives like Resorcinol Formaldehyde Latex (RFL) and isocyanates which form as an interfacial layer leading to good adhesion with the rubber matrix through chemical groups reactions. However as nylon fibre remain circular and smooth, by virtue of manufacturing through melt spinning, the mechanical interlocking effect with the matrix remain poor. Therefore, surface roughening of nylon fibre would improve the interfacial adhesion. Exclusive study of the effect surface modification on the interfacial adhesion properties of nylon and rubber composites are very limited.

Therefore, the present study aims to develop pre-treatment processes for surface modification the polyamide fabric to increase the interfacial adhesion strength of rubber composite. For the purpose, nylon 6,6 was treated with three different pre-treatment processes such as formic acid, protease enzyme and low temperature plasma (LTP) treatments to modify the surface. Initially, the treatment conditions such as concentration, time, temperature and exposure time are optimized. Physico-chemical surface roughening of nylon obtained through three different processes are characterized using scanning electron microscope (SEM), geometrical surface roughness (SMD) through KES-FB4 surface tester, Fourier Transform Infrared spectroscope (FTIR) and differential scanning calorimeter (DSC), Xray diffraction (XRD) and tensile strength testing.

Control and surface modified nylon fabrics are then subjected to RFL (Resorcinol Formaldehyde Latex) treatment for making rubber composites. The RFL add-on was maintained at 12% of the fabric weight for all samples. The treated fabrics are dried at 100°C and cured at 160°C for 3 min. Rubber composite samples were prepared in a mould with dimensions of (150 mm x 150 mm x 6 mm). First, the mould was pre-heated to 150 \degree C and then the bottom half of the cavity was filled with 3 mm thick rubber sheet. The polyamide fabrics were then placed over the rubber sheet in the mould. Finally, the remaining half was filled with rubber sheet and cured at 150°C for 10 min at a pressure of 200 Kg/cm^2 . The cured samples were conditioned at ambient temperature for 24 hrs before carrying out the testing. Further, the samples, thus prepared with control and surface modified nylon 6,6 were subjected to peel strength testing as a measure of the interfacial bonding between the fabric and the rubber.

All the three methods of treatments had an optimum treatment conditions at which the maximum surface roughness effect and consequent high peel strength were noticed. Beyond such treatment conditions, the

surface roughness started decreasing primarily because of the pore merging effect. The optimum conditions for formic acid treatment were 11.2% formic acid concentration, 70°C temperature and 30 min; the conditions for protease enzyme treatment were enzyme concentration 3%, time 60 min, temperature 70 °C and the condition for LTP treatment was 180 s treatment time. Under the optimum conditions, the SMD values were 20.1 μ m, 20.3 μ m and 20.3 μ m for the formic acid, protease enzyme and plasma treatment methods, respectively. The peel strength improvement for the rubber composite in the formic acid treatment was 150 % and 124 % for warp and weft directions, respectively; in the protease enzyme treatment was 145 % and 114 % for warp and weft directions, respectively and in the LTP treatment was 150 % and 122 % for warp and weft directions, respectively.

SEM analysis of all three pre-treatments confirmed the presence of heterogeneous appearance like cracks, etches and micro-rough surfaces on the treated Nylon-6,6 Fibre surface. The pores found for the optimum treatment conditions had average pore size of about 100 nm $(0.1 \mu m)$. SEM analysis of the interface of the samples after peeling showed the presence of intense deposits of rubber particles on the treated samples while the deposits were only a few on the control samples.

Physico-chemical mechanisms of roughening with pore merging effect for the treatment processes through acid, enzyme assisted hydrolysis and plasma species were proposed. FTIR analysis showed the changes in chemical groups as a result of the treatments. Other studies such as DSC, XRD and tensile strength testing results showed that there were no notable differences compared to the control samples indicating that the treatments did not influence the bulk properties practically.