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Adsorption Studies of Ca^{2+} Ions on α-Fe_2O_3 Dispersed Natural Rubber Composite: Structure, Morphology and Thermal Studies

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Ultrafine particles of α-Fe_2O_3 dispersed natural rubber composite film prepared by solvent casting method are reported. The prepared film is subjected to Ca^{2+} ions adsorption. The adsorbent is characterised with XRD, SEM and thermal studies. The eluent is estimated for Ca^{2+} ions by EDTA complexometric titration and the results are compared before adsorption. The solution state conductivity shows increase in conductivity due to decrease in concentration of Ca^{2+} ions.

Key Words: Adsorption, Ca^{2+}, α-Fe_2O_3 Structure, Morphology.

INTRODUCTION

Polymer composites containing ferrites are increasingly replacing conventional ceramic magnetic materials, because of their mouldability and reduction in cost. They are also potential materials as microwave absorbers and other aerospace applications. These flexible magnets or rubber ferrite composites are possible by the incorporation of magnetic powders in various elastomer matrices. The addition of magnetic fillers in an elastomer matrix modifies to physical properties of the matrix considerably[1–8]. They modify the dielectric properties and impart magnetic properties to the matrix. Appropriate dielectric constant and suitable magnetic permeability is essential for application at high frequencies and this is possible through rubber ferrite composites. This report also shows that non-magnetic nanocomposites are possible by the chemical incorporation of ferrite particles in polymer matrix. Understanding the chemistry of these composites is very important for correlation of properties and tailoring materials for specific applications. Hence this paper reports α-Fe_2O_3 dispersed natural rubber for its synthesis, structure, thermal properties and morphology.

EXPERIMENTAL

Purification of α-Fe_2O_3: Minimum amount of commercially available red-oxide (IS-445) was dissolved in hydrochloric acid and was then reprecipitated by adding liquid ammonia solution (1 : 1). The precipitate was filtered, washed and dried. The whole compound was transferred to a clean crucible and heated in an electrical oven until the red [above 600°C] coloured powder was obtained.

Preparation of α-Fe_2O_3 Composite: A known weight of natural rubber was dissolved in xylene solvent. To this 5 weight % of α-Fe_2O_3 was added and sociated for 1 h. After substantial mixing of the α-Fe_2O_3 the solvent was casted on a hot plate; a uniform dispersed α-Fe_2O_3 rubber film was used for adsorption studies.

Adsorption of Ca^{2+} ions onto the composite: In a 500 mL beaker known quantity of α-Fe_2O_3 dispersed rubber composite was taken and to this 100 mL of 0.01 N $CaCl_2$ solution was added. The mixture was kept on a mechanical stirrer for 12 h. After 12 h the solution was decanted and tested for Ca^{2+} ions by EDTA titration. The composite was characterised by X-ray diffraction (XRD), scanning electron micrography (SEM) and thermal studies (TGA/DTA).

RESULTS AND DISCUSSION

The adsorption of Ca^{2+} ions onto the composite films was tested by various methods including EDTA titration of Ca^{2+} ions before and after adsorption. The preliminary results by titrations indicated decrease in Ca^{2+} ions on adsorption by the composite film compared with original $CaCl_2$ solution.

X-ray diffraction studies (XRD): Fig. 1* shows the XRD pattern of as synthesised α-Fe_2O_3 (purified). All the peaks are indexable and matching with JCPDS file No. 24–81. No other peaks are found as impurities.

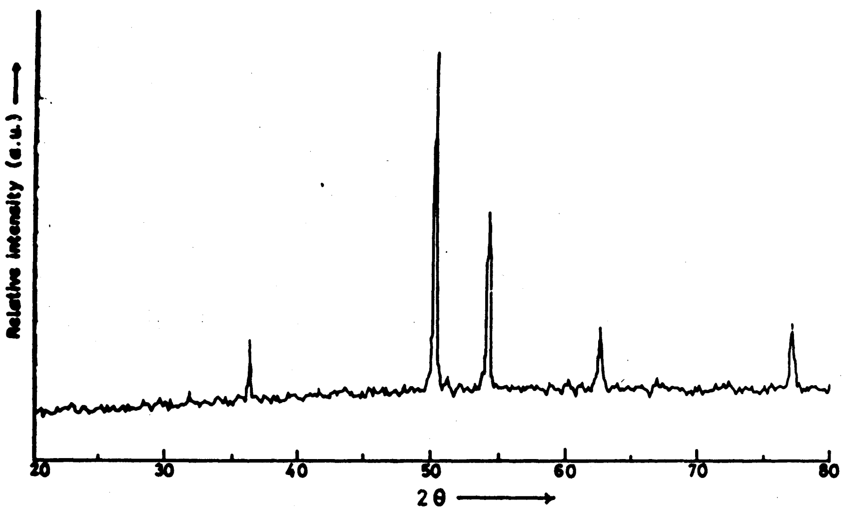

Fig. 1. XRD pattern of α-Fe_2O_3 (VH_1 sample)

Fig. 2 shows the XRD pattern of Ca^{2+} ions adsorbed α-Fe_2O_3 dispersed natural rubber composite. The XRD pattern indicates dominant presence of crystalline rubber phase rather than α-Fe_2O_3 phase. The explanation for this is that the majority of the polymers in the α-Fe_2O_3 particles are embedded uniformly throughout the matrix; hence, decrease in degree of crystallinity of α-Fe_2O_3 when compared with major polymers. We observe that only Bragg's reflection corresponding to crystalline polymers rather than α-Fe_2O_3.

Morphology: Fig. 3 shows the SEM image of as prepared α-Fe_2O_3. From the image one can see clearly that particles are in the range 100–300 nm. We can also see various shapes and sizes of the particles. One important feature is that there is no aggregation of particles to form giant structures; instead, all particles are individual (Fig. 3).

Fig. 4 shows the SEM image of natural rubber. As expected, rubber shows some

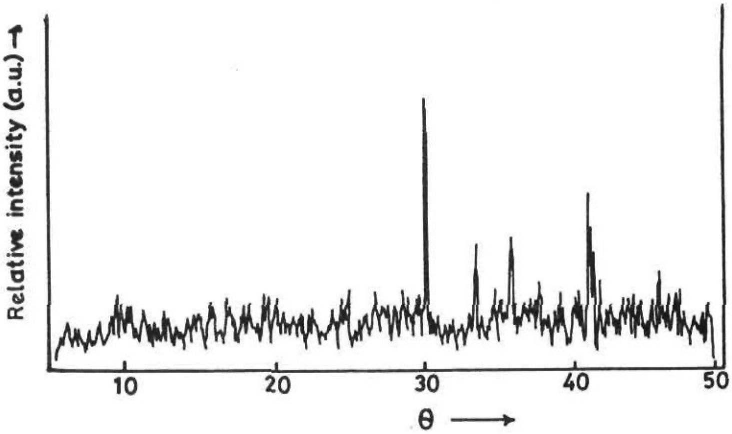

Fig. 2. XRD pattern of Ca^{2+} ions adsorbed α-Fe_2O_3 dispersed natural rubber composite (VH_2 sample)

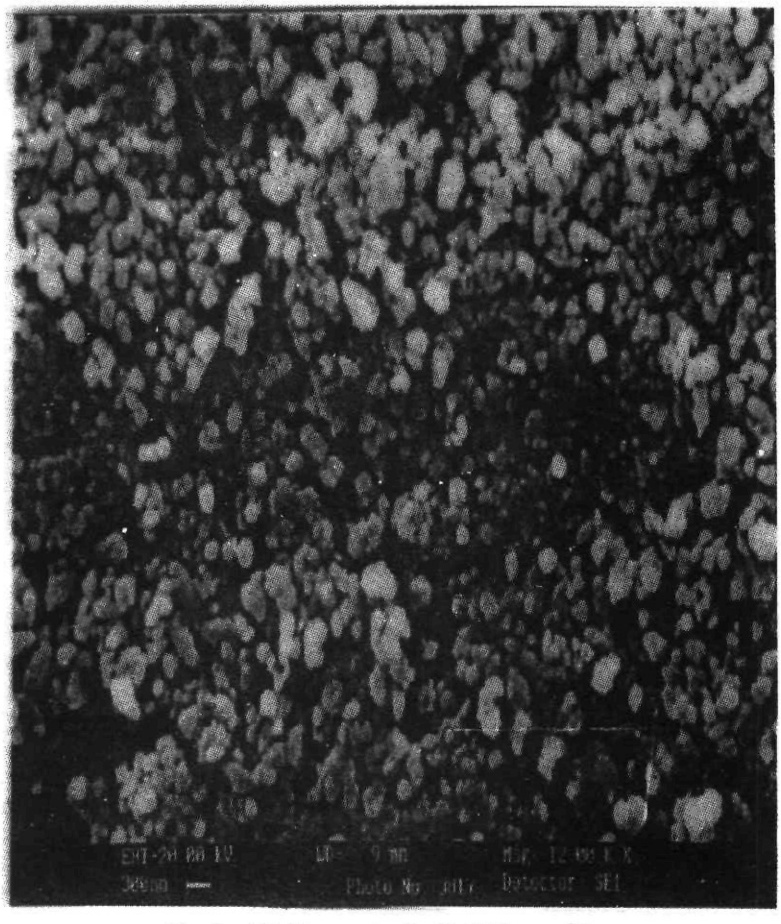

Fig. 3. SEM image of α-Fe_2O_3 (VH_1 sample)

stretched kind structure. Fig. 5 shows the SEM image of Ca^{2+} ions adsorbed on α-Fe_2O_3 dispersed composite film. From the figure, it is clear that the Ca^{2+} ions are adsorbed onto the composite film. The ball-like structure corresponds to Ca^{2+} ion on the surface of the composite film.

Fig. 4. SEM image of natural rubber

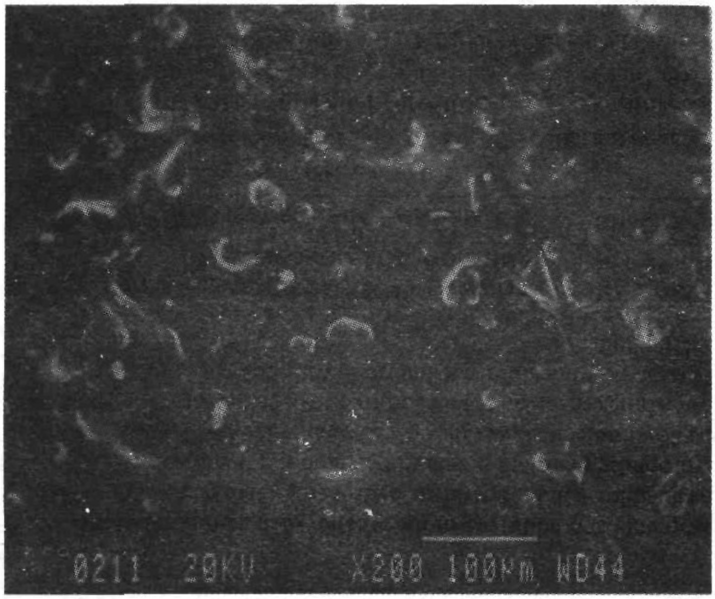

Fig. 5. SEM image of Ca^{2+} adsorbed on α-Fe_2O_3 dispersed natural rubber (VH_2 sample)

Thermal studies: Fig. 6 shows the thermal trace of Ca^{2+} ions adsorbed α-Fe_2O_3 dispersed natural rubber composite. From the figure it is clear that the composite film started decomposing from 290°C and ends at 400°C, but in case of pure rubber it was 280°C. The explanation given for this is that the dispersion of α-Fe_2O_3 in the rubber matrix increases the thermal stability of the polymer and also because of Ca^{2+} ions adsorption. In DTA we can see the phase transformation of α-Fe_2O_3, since α-Fe_2O_3 exists in different crystal forms.

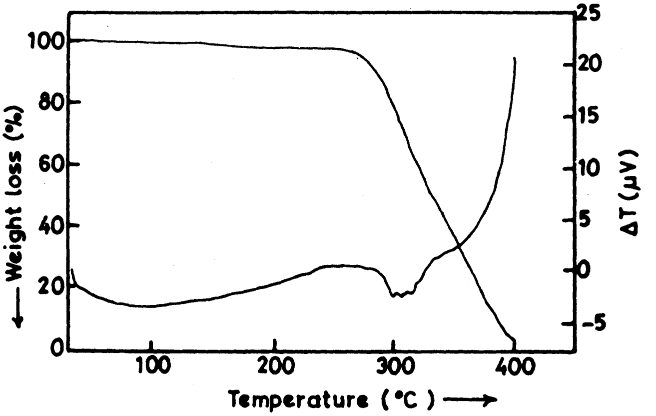

Fig. 6. Thermal (TG/DTA) trace of Ca^{2+} adsorbed on α-Fe_2O_3 dispersed natural rubber (VH_2 sample)

Conclusions

- A uniformed α-Fe_2O_3 dispersed natural rubber composite is prepared and characterised for its structure and morphology.
- SEM studies show the adsorption of Ca^{2+} ions onto the composite films.
- Addition of α-Fe_2O_3 into the polymer matrix increases the thermal stability of the natural rubber.
- The α-Fe_2O_3 dispersed natural rubber composite films can also be used to study the adsorption behaviour of other transition metal ions.

REFERENCES

1. J. Richard, P. Spontak and Nikunj P. Patel, *Current Opinion in Colloid & Interface Science*, **5**, 334 (2000).
2. H.O. Jungk and C. Feldmann, *J. Mater. Res.*, **15**, 2244 (2000).
3. M. Zdujic, C. Jovalekic, Lj. Karanovic, M. Mitric, D. Poleti and D. Skala, *Materials Science and Engineering*, **A245**, 109 (1998).
4. J.E. Mark, *Current Opinion in Solid and Materials Science*, **4**, 565 (1999).
5. K. Yuewkli, R. Krishnamoorti, M.F. Tse, K.O. Mcelrath, A.H. Tsou and H.C. Wang, *J. Polym. Sci.-B, Polym. Phys.*, **39**, 256 (2001).
6. B.H. Sohn and R.E. Cohen, *Chem. Mater.*, **9**, 264 (1997).
7. M.P. Won, B.C. Dave., F. Leroux., J. Harreld, B. Dunn and L.F. Naz, *J. Mater. Chem.*, **8**, 1019 (1998).
8. K.B. Cheng, S. Ramakrishna and K.C. Lee, *Composite*, 31A, 1039 (2000).

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