CHAPTER 9

SUMMARY AND CONCLUSION

The objectives of the present work are to develop hybrid matrix materials and nano hybrid composites based on industrially valuable polymeric intermediates viz., epoxy resin, polysulfones and bismaleimides and their characterization by different methods. The hybrid bismaleimide modified polysulfone-epoxy matrix systems were developed using polysulfone/polyethersulfone and bismaleimides namely N,N'-bis(maleimido)4,4'-diphenylmethane (BMI-1), 1,3-bis(maleimido) benzene (BMI-2), 3,3’-bis(maleimidophenyl)phenyl phosphine oxide (BMI-3) and 1,1’-bis(4-maleimidophenyl)cyclohexane (BMI-4) which were used as chemical modifiers for epoxy resin.

The role and importance of epoxy resin, curing agents, toughening of epoxy resin with high performance thermoplastic modifiers like polysulfone, bismaleimides, fibre reinforced composites and organo clay filled nanocomposites, have been reviewed in Chapter 1.

Synthesis of bismaleimides along with preparation of polysulfone modified epoxy, polyethersulfone modified epoxy, bismaleimides (BMI-1, BMI-2, BMI-3 and BMI-4) modified epoxy, bismaleimides modified polysulfone-epoxy and bismaleimides modified polyethersulfone - epoxy hybrid matrices was explained in Chapter 2.
The procedure for the preparation of organophilic montmorillonite clay was also discussed in Chapter 2. In addition, the experimental procedures for the characterization of physico-chemical, mechanical, electrical, thermal, dynamic mechanical properties and morphological analysis of matrix systems and nanocomposites were presented in Chapter 2.

The formation of network between bisphenol-A based polysulfone and DGEBA based epoxy resin was confirmed by FTIR spectra. The structure and the functional groups terminated bismaleimides synthesized in the present work were ascertained by FTIR and NMR characterization. The homopolymerization of bismaleimides (BMIs) in the presence of epoxy resin occurred at much lower temperature (130-140°C) than expected due to Michael addition reaction which was confirmed by FTIR spectra. This may be explained as due to the formation of zwitter ion adduct between the oxirane ring of epoxy resin and the electron deficient BMI double bond, which anionically induce the homopolymerization reaction of bismaleimides (BMI-1, BMI-2, BMI-3 and BMI-4) at lower temperature (130-140°C) in the presence of epoxy resin, whereas it occurs normally at about 180°C, 202°C, 199°C and 223°C respectively.

The mechanical properties of unmodified epoxy, polysulfone modified epoxy, bismaleimides modified epoxy and bismaleimide modified polysulfone/polyethersulfone epoxy matrix materials and organo clay filled nanocomposites were studied as per ASTM standards. The study indicated that the polysulfone incorporation into epoxy resin increased the tensile and flexural properties to a marginal extent. This may be explained as due to inherent rigid aromatic molecular structure and higher elastic modulus behaviour imparted by polysulfone.
The tensile and flexural properties of both epoxy and polysulfone-epoxy systems were found increasing with the incorporation of bismaleimides (BMI-1, BMI-2, BMI-3 and BMI-4) according to their nature and percentage concentrations. This is due to the formation of intercross-linking network between bismaleimides and epoxy systems, which inturn enhanced crosslinking density and rigidity. Among bismaleimides modified epoxy and polysulfone epoxy systems, the BMI-2 modified systems showed higher improvement in both tensile and flexural properties than BMI-1, BMI-3 and BMI-4 modified systems. This observation may be due to low molecular weight and the higher number of reactive sites available in the case of BMI-2, which inturn increased the crosslink density and rigidity when compared with those of BMI-1, BMI-3 and BMI-4 molecules.

The impact strength of epoxy resin was increased with the incorporation of polysulfone according to its percentage concentration. This is due to the chain lengthening by the introduction of polysulfone and formation of IPN structure within the system. Further the presence of ether linkage in the polysulfone skeleton also responsible for enhancement for values of impact strength due to its free molecular rotation. Impact strength of epoxy and polysulfone-epoxy system was decreased with increasing bismaleimides concentration. This can be explained due to the formation of intercross linking network between bismaleimides and epoxy systems, which inturn increases the rigidity. The reduction in the value of impact strength is higher in the case of BMI-2 modified systems than BMI-1, BMI-3 and BMI-4 modified epoxy systems.

The electrical insulation behaviours like dielectric strength, volume resistivity, surface resistivity and arc resistance of polysulfone modified epoxy systems were decreased with the increasing concentration of polysulfone due to the presence of polar nature of sulfone and ether linkages.
The electrical insulation properties were enhanced by incorporation of bismaleimides according to their concentration of both unmodified epoxy and polysulfone modified epoxy systems. This can be attributed due to the improved tendency to resist moisture absorption.

The cure reaction behaviour epoxy-DDM, polysulfone modified epoxy–DDM and bismaleimides modified epoxy systems were studied by DSC. From the results, it can be ascertained that all hybrid epoxy systems follow unimodel reaction behaviour. The increase in the peak maximum temperature with increasing bismaleimides concentration for BMI-1, BMI-2, BMI-3 and BMI-4 modified epoxy systems confirmed that the homopolymerization reaction of bismaleimides predominated over Michael addition reaction. The glass transition temperature (Tg) of epoxy modified bismaleimides (BMI-1, BMI-2, BMI-3 and BMI-4) is enhanced due to the reaction of homopolymerization rather than Michael addition.

The incorporation of polysulfone into epoxy resin has a significant effect on Tg. The incorporation of polysulfone raised the viscosity of the system, which could result in an incomplete curing reaction due to the steric hindrance under the curing conditions. The thickening effect of polysulfone is also an important factor which gives rise to the reduction in the crosslinking density and consequent chain lengthening of polysulfone skeleton and hence molecular flexibility occurs at low temperature which leads to reduction in the values of Tg.

The value of Tg of epoxy and polysulfone-epoxy systems were increased with increasing percentage incorporation of BMI-1, BMI-2, BMI-3 and BMI-4. The increase in Tg values for BMI-1, BMI-2, BMI-3 and BMI-4 modified epoxy and polysulfone- epoxy systems was due to the homopolymerization reaction of bismaleimides rather than Michael addition.
reaction. Michael addition reaction led to the formation of thermally weak amine linkage and ultimately reduced the crosslink density due to chain extension. Further the improvement in Tg lends to support to the earlier observation made by Musto et al 1998, that in the presence of epoxy resin the homopolymerization reaction of bismaleimides starts even below at 130°C. The single Tg value obtained for all bismaleimides modified epoxy and polysulfone epoxy systems confirmed the formation of intercross linking network and the hybrids are single chemical entity.

The incorporation of polysulfone into epoxy resin improved thermal stability and enhanced degradation temperature according to its percentage concentration. The presence of polysulfone skeleton in the epoxy system delays the degradation process and high thermal energy is required to attain the same percentage weight loss than that required for unmodified epoxy system. Similarly the bismaleimides incorporation into epoxy and polysulfone epoxy system also enhanced thermal degradation temperature and this may be due to the formation of intercross linking network between epoxy and bismaleimides and also due to the presence of rigid heterocyclic ring structure of bismaleimides. The flame retardancy of bismaleimide modified polysulfone epoxy systems increases significantly according to its percentage concentration. Among the bismaleimides, the phosphorous containing bismaleimide (BMI-3) modified systems execute better flame retardancy due to the formation of high char yield than others.

The values of heat distortion temperature (HDT) of epoxy resin were decreased with increasing polysulfone concentration. However, HDT values of bismaleimides (BMI-1, BMI-2, BMI-3 and BMI-4) incorporated epoxy and polysulfone-epoxy systems were enhanced according to the percentage concentration of bismaleimides.
The surface morphology of unmodified epoxy system was characterized by the scanning electron microscope (SEM), which showed a smooth, glassy and homogeneous microstructure. The micrograph of fractured surfaces of polysulfone epoxy systems and bismaleimides modified epoxy systems was almost similar to that of unmodified epoxy system. This indicates that there is no separate phase domains of the two components involved and also suggests the formation of homogeneous intercrosslinked network.

Polysulfone and bismaleimides incorporation into epoxy resin decreased the water absorption behaviour, which may be due to the hydrophobic behaviour imparted by polysulfone and negligible water absorption behaviour of bismaleimides.

The organo clay filled epoxy and polysulfone-epoxy-nanocomposites were prepared and the formation of nanocomposites was ascertained from XRD and SEM studies. The nanocomposites exhibited a significant improvement in thermomechanical properties and negligible tendency to water absorption than that of the unmodified and unfilled epoxy systems.

The data obtained from different studies with varying types of matrix systems developed in the present investigation namely, polysulfone modified epoxy, bismaleimides modified epoxy, bismaleimides modified polysulfone epoxy matrix systems and organo clay filled modified epoxy nanocomposites, it can be concluded that the BMI–2 modified polysulfone–epoxy system has exhibited better impact strength, thermal stability, better weathering resistance with good electrical insulation characteristics than other systems developed and it can effectively be used for the fabrication of advanced aerospace hardware composites components, besides find use for
the manufacture of products for high performance dielectric and industrial components to be used under adverse environments and high temperature service conditions. Further the organo clay filled epoxy polysulfone nanocomposites also exhibit better thermomechanical properties than unfilled counterparts and could be used as effective material in the form of adhesives, sealants and matrices for varying industrial and engineering applications for better performance with improved longevity.