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MODIFIED THERMOSETS – A REVIEW

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ABSTRACT

Recent and extensive reports on the development of preparation methods that can lead to obtaining various nanostructured polymeric materials, including the preparation of nanoparticles, nano-capsules, nano-gels, nano-fibres, dendrimers and nano-composites, have been presented in the literature to provide a picture of the complexity of the field, and the diversity of approaching methods. From the data presented by various researchers, it appears that by ensuring a rigorous control of the nano-structuration of polymers and/or by adding nano-particles to the polymer matrices, improvements in structural and functional properties can be achieved in a significant number of polymer systems as a response to the continuous demands of industrial advanced sectors. Until now, most of the studies on polymer blends have been related to the control of their physical and chemical properties, their barrier properties or their electrical conductivity. This paper briefly presents various applications of resins (thermoset polymers) used to synthesize some polymer materials and blends.

KEYWORDS: nano-fibres, thermoset polymers, synthesis

1. Introduction

Miscible polymer blends offer great design capability over shape memory effect (SME) with controllable mechanical properties and stimuli sensitivity by simply changing the constituent compositions. However, understanding the thermodynamics behind those IMMs on miscible polymer blends has not yet been explored [1-3]. For the advancement of polymer science and polymer technologies, the chemical characterization of the polymer mixture remains the fundamental analytical study [4-6]. Mixing polymers and inorganic nano-materials opens new paths for synthesizing flexible composite materials and improves the macroscopic performance of the materials [7-9]. Polymers can be in raw form (filaments, powders, resins and reactive agents) for various categories of use [10].

Recently, increased attention has been imposed on the environment, what causes an impediment to the use of thermosetting resins [11-13]. Thermosetting resins are known to be cheaper in terms of processability compared to thermoplastics, which require high production costs and higher energy consumption [14-16]. Even though thermoplastic resins have advantages such as: recyclability, thermoformability and weldability, they have not yet reached the same technological maturity as thermosetting resins. Thermoplastics have the ability to form liquids during the heating process and become hard when the cooling process takes place [17-21].

2. Epoxy resins (ERs)

Epoxy resin is considered one of the most important thermoset polymers, having in its composition at least epoxy groups in the molecule. It comes in three forms: liquid, solid or viscous [22-24]. According to the latest reports, epoxy resins currently have the highest sales percentage (about 70%) in the thermosets market, the main advantages being excellent mechanical properties, thermal stability, outstanding electrical and chemical insulation and low cost [25-32]. Epoxy resin (EP) is most often used in the electronic and electric field (design of tiny electronic components, generators, motors, voltage distribution networks or electrical insulating materials) [33-37]. Due to the poor weathering resistance and high brittleness of epoxy resin, it is necessary to improve the performance of the EP matrix to meet the requirements of composite materials [38-44]. Epoxy resin can be modified by both chemical and physical methods. According to studies in the literature, chemical modification involves quite high costs and some deficiencies of the complex technology [45-47]. On the other hand, physical modification of EP is the most used method. The main types of modifiers are inorganic nanoparticles, rubber particles and thermoplastics [48-50].

3. Polyurethane resins (PUs)

Polyurethanes are a class of polymers that exhibit three types of behaviour: thermoplastic, thermoset, and elastomer. PUs present versatile properties both thermally and in terms of mechanical and chemical aspects [51]. Compared to epoxy resins, PUs shows better fatigue and damage resistance properties. At the same time, they have the advantage of being synthesized with various vegetable oils to replace petrochemical products with renewable or recycled materials [52-54]. In most cases, polyurethanes are used for sealing, or for protection against condensation [55, 56]. Polyurethanes belong to the category of foaming materials, with high porosity, light weight and good energy absorption capacity, being excellent for packaging and damping sensitive objects [57-59]. Linear polyurethanes have good toughness and re-processability, but have poor solvent resistance [23, 60-62].

4. Vinylester resins (VER)

Vinylester resins (VERs) are used in various commercial applications (automotive parts, coatings, adhesives, military and aerospace applications, moulding compounds) due to their high hardness, low exothermic heat, low volume shrinkage and low cost [39, 63-67]. Nature provides us with a variety of plant biomass that we can use as raw material for the production of precursors and chemical substances (plant oils, tung oil, cashew nut shell liquid (CNSL), lingo-cellulosic, fatty acid dimers) for new VERs [68-72]. Materials containing VE can be used as neutron shielding material for radioactive material transport/storage containers [73]. Fibre reinforced plastic (FRP) manufacturers are using vinylesters because they offer greater resistance to attacks in aggressive chemical environments, or other types of acids and caustics [72, 74, 75].

5. Thermosets and thermoplastics applications

Yan Wang and his collaborators [76], developed a multi-scale simulation scheme by which they generated the atomic structure of epoxy resins. The simulation scheme they proposed provides a reasonable use of the advantages of the DFT method in dealing with systems involving the breaking and formation of chemical bonds. This multi-scale simulation strategy provides a possible investigation scheme to study such an epoxy resin system, which can also be applied to study other similar composite materials with complex network structures.

Ruowen Tu and Henry Sodano [72] fabricated high-performance vinylester resin-based nano-composites by direct ink writing with dual UV-thermal curing. The fracture strength of dual-imprinted and reinforced VER nano-composites was also 16% higher than MEKP-reinforced pure cast VER.

In [77], the authors synthesized the organo-silicon additive-modified epoxy resin RSN-6018 through the condensation reaction between the organo-silicon intermediate (RSN-6018) and the epoxy resin (E-20). The results of mechanical property testing showed that the incorporation of RSN-6018 organo-silicon improved the hardness of the cured ER films.

Apriany Saludung and his collaborators [78] incorporated epoxy resin into the geopolymer matrix. The improvement of mechanical strengths and alkali binding properties of naturally hardened geopolymer by adding epoxy resin can lead to extensive application of geopolymer binder in the construction industry.

In [39], researchers created a high-performance permeable concrete with polyester and epoxy resins. From the tests carried out, they concluded that: increasing the resin content improves the compressive, tensile and flexural strength, while an increase in the size of the coarse aggregate and the use of epoxy resin instead of polyester resin decreases the previously listed strengths.

Valentino R., *et al.* [57], carried out an experimental study on the mechanical behaviour of two polyurethane resins used for geotechnical applications. Experimental measurements showed that resin density and confining pressure are closely related, and for the same type of resin under limited conditions, the mechanical response is constant.

Peng Y.-J., *et al.* [51], designed a composite material consisting of polyurethane and epoxy resin with multi-level structure and high performance. Such a well-established structure–property relationship could further provide guidance for the fabrication of high-performance recyclable material with precisely controllable microstructures and behaviours.

In [32], the authors studied the changes in structure, mechanical and adhesion properties of vinylester resins after aging. Chemical and structural changes were observed in vinylester resins after aging in a climatic chamber. Most changes occur in the first three days.

Banna M. H., *et al.* [63], analysed the effects of two acidic aqueous solutions on polyester and bisphenol A epoxy vinylester resins. For both resins, the average hardness increased more after two weeks of exposure and then decreased after four weeks of exposure due to a relationship between hardness, micro-hardness indentation depth, and microstructure. Acid exposure affected the polyester resin more than the bisphenol epoxy vinyl ester.

Yang X., *et al.* [80], produced a type of solvent-free epoxy-modified silicone resin transparent coating with high transparency and low volume shrinkage. The silicone layers that were obtained have a transmittance greater than 95% and a hardness of 5H after UV curing with a percentage of 3% by mass for 20 s time interval. It was found that the silicone resin materials obtained have good thermal stability, a good adhesion and a fairly low volume shrinkage.

In [64], researchers improved the thermal, impact and tensile properties of unsaturated thermosetting polyester (UP) by blending with thermosetting vinylester (VE) and methyl methacrylate (MMA). UP blended with 30%VE and 10%MMA had the highest impact strength (an increase of 17.6%), higher tensile strength (an increase of 45.5%) and higher elongation at break (an increase of 26.9%) compared to pure UP. The addition of VE and MMA to UP led to an increase in the toughness of UP as a result of disruption of the chain network links of UP, resulting in a decrease in structural stiffness, and an increase in the plastic strain fraction.

The enhancement of solvent resistance and thermo-mechanical properties of thermoplastic acrylic polymers and composites by reactive hybridization has been studied [81]. Both unreinforced polymer blends and glass fibre reinforced composites have been studied. The authors' findings showed that incorporating reactive PPE into a reactive acrylic resin to produce a hybrid matrix system is a simple and effective strategy for increasing solvent resistance.

Luo D., *et al.* [82], incorporated Fe_3O_4 and SiO_2 nano-particles into epoxy modified silicone resin (ESR) as a coating for soft magnetic composites (SMC) with improved performance. On the one hand, the introduction of Fe_3O_4 reduces the magnetic dilution effect, which is beneficial for magnetization and permeability. On the other hand, the incorporation of SiO_2 prevents the agglomeration of Fe_3O_4 nano-particles and leads to an increased electrical resistivity as well as increased mechanical strength of SMCs.

Wei Y.-Y., *et al.* [83], fabricated both photo-polymerized and thermo-polymerized silicone hydrogels with different surface microstructure and wettability. It was found that the surface of silicone hydrogels prepared by thermal polymerization was significantly more hydrophilic than the surface made by photo-initiation polymerization. ATR-FTIR and SEM as well as XPS analysis indicated that the chemical composition and elemental distribution of the two hydrogels were similar. However, AFM images showed that the silicone hydrogel prepared by photo-polymerization has a lot of tiny pores on the surface, so air bubbles can be stored. Whereas, the surface of silicone hydrogel obtained by thermal polymerization has some tiny slopes.

In [84], researchers devised a method by which they recycled waste thermoset unsaturated polyester resins into oligomers for the synthesis of amphiphilic aerogels. Styrene-maleic acid copolymer (SMC) was obtained by cleavage of ester groups from waste thermoset unsaturated polyester resins (WTUPR). The degradation occurred at 180 °C in glycol with potassium carbonate as a catalyst for 5 h, and the resulting potassium salt of SMC (SMC-K) could be very easily separated by precipitation using ethanol with a yield of 63.8%. Good compressibility and strength of the aerogel were demonstrated.

Zheng C., *et al.* [85], developed a negative ion implantation system to study the surface modification of room temperature vulcanizing silicone rubber (RTV SR) for cell biocompatibility. They concluded that the main reason for the improved hydrophilicity is that the implanted ions result in a new surface atomic bonding state and a new morphology. Based on the XPS and ATR-FTIR results, it can be deduced that ion implantation breaks the hydrophobic functional groups of RTV SR and generates functional groups. SEM demonstrated that cracks appear on the implanted surface of RTV SR.

Sultania M., *et al.* [71], conducted studies on the synthesis and curing of epoxidized novolac vinyl ester resin from materials from renewable resources. It was concluded that the synthesis of low viscosity vinylester resin from renewable resource materials such as cardanol could reduce the use of harmful chemicals such as styrene during curing of such resin. The cure temperature was found to be very close to the cure temperature for VER of epoxidized phenolic novolac resin cured using 40% styrene. The curing time, using 40% styrene, for the CNEVER sample was lower than for the PVER sample. Also, the curing time is the shortest when the CNEVER sample is used without styrene. Undoubtedly, the cost of the resin will be lower than that of already existing VERs.

Malucelli G., *et al.* [54], synthesized adhesives based on polyurethane resins. They investigated the hardening reaction and the influence of surface properties on adhesion. Curing kinetics of the PU resin, evaluated by FTIR spectroscopy, revealed an asymptotic trend, with a reaction completed after approximately 100 h. The results of the adhesion measurements of the PU adhesive on different substrates were in total agreement with the surface properties of both the resin and the substrates.

Cristiana Ittner and Maria Felisberti [65], studied the influence of three silicone-based additives to modify the properties of vinylester resin. The study of the curing kinetics revealed that these additives influenced the curing kinetics. The influence of these additives is directly related to the initiator/catalyst/promoter system used in curing, which can accelerate or delay the reaction depending on the presence or absence of DMA. The use of DMA as a peroxide decomposition promoter in the initial stage of resin curing in the presence of silicone-based additives results in an increase in the reaction enthalpy and a decrease in the activation energy. This suggests that the curing mechanism is modified due to silicone-based additives.

Zhao H., *et al.* [86], analysed the curing kinetics of four epoxy resins using a diamine-terminated polyether as curing agent. It has been shown that a diamine-terminated polyether (DAPE) can enhance the flexibility of epoxy systems, which is attributed to the unique chemical structure. The results showed that the curing reaction can be described by the Sestak-Berggren (SB(m,n)) kinetic model and fits well the experimental curves. According to the tensile test and DMA test results, the strengthening effect of DAPE was successfully achieved on four epoxy systems. The cure is modified due to the silicone-based additives presence.

Pragnesh Dave and Nikul Patel [87], prepared unsaturated poly(ester-amide) resins based on epoxy resins (UPEA). These UPEAs were then treated with acryloyl chloride to obtain acrylated UPEA resin (ie AUPEA). Interacting mixtures of equal proportions of AUPEA and vinyl ester epoxy resin (VE) were prepared. The general advantages of the produced PEA formation are as follows: the intermolecular reaction with bismaleimides formed UPEA with good chemical resistivity and moderate thermal stability. The glass fibre reinforced composites of all PEAs were laminated and exhibited excellent strength properties and good mechanical and electrical properties.

In [88], a silicone-epoxy copolymer was prepared by synergizing the o-allylphenol-modified (UC-233) and methoxy-terminated silicone methylphenyl intermediate (PMPS-S). SEM and DMA revealed that the comb-shaped silicone-epoxy copolymer showed better compatibility due to the pre-chemical grafting reaction. In addition, TGA showed that the thermal stability of the comb-shaped silicone-epoxy copolymer was improved and maintained the excellent mechanical properties of the epoxy resin. TGA-FTIR shows the thermal degradation process that methoxy-terminated methylphenyl silicone intermediate (PMPS-S) and phenyl hydrogen-containing silicone oil (UC-233) could inhibit the degradation of epoxy resin.

In [67], the synthesis and the investigation of a new brominated vinyl ester resin are presented. The DMA results demonstrate that all resin formulations comprising Br-VER exhibit improved mechanical performance compared to the commercial vinylester resin. Rheological studies on the resin compositions showed that, compared to the commercial resin (Derakane 510A-40), the synthesized Br-GVER has a significantly lower viscosity and a longer lifetime (9 hours vs. 2 hours), thereby eliminating the need for an inhibitor when used for applications such as resin infusion.

Studies have been conducted on the preparation and super-hydrophobic surface properties of RTV silicone rubber [89]. The authors concluded that the preparation of the super-hydrophobic surface by physical deposition has the advantages of simple processing, low cost and large surface area preparation. In addition, SR has outstanding high temperature resistance. Therefore, this super-hydrophobic material is suitable to be applied at both room temperature and high temperature, and has wide-scale application prospects.

In [90], a study was conducted on the modification of room temperature vulcanized silicone rubber (RTV) by microencapsulated phase change material (MEPCM). MEPCM blended RTV has been shown to have better thermal insulation than regular silicone rubber. As the mass ratio of MEPCM increases, it takes more time for the modified RTV to cool to 0 °C. The decrease in volume resistivity of the modified RTV can increase the leakage current, which is also a good aspect for anti-icing.

Goram Gohel and his collaborators [91], used epoxy resins and carbon fibres to manufacture sports helmets. Overall, detailed manufacturing and certification tests performed on helmets have shown significant potential for using the composite shell as a viable alternative to conventional helmet material systems in terms of achieving increased safety.

In [92], researchers studied the effects of graphene on various properties and applications of silicone rubber and silicone resin. They found that graphene has high electron mobility, thermal conductivity and mechanical properties at room temperature. As a typical carbon nano-scale filler, graphene added to the polymer matrix can improve the performance of the polymer.

Vincent Joseph and collaborators [93], combined rigid epoxy resins with platinum-catalysed soft silicone rubbers for additive manufacturing of soft robots. By adjusting the composition of the hybrid resins, the elastic modulus can be tuned over an unprecedented ratio of five orders of magnitude (from 20 kPa to 2 GPa) with remarkable interfacial strength (from 1 to 3 kJ m^{-2}). The significance of this new class of hybrid resins is demonstrated by the fabrication of various functional devices relevant to wearables, healthcare and soft robotics. Overall, these hybrid resins open new frontiers for manufacturing the next generation of soft robots using unique continuous additive manufacturing processes.

In [94], researchers synthesized and characterized new room temperature vulcanized (RTV) silicone rubbers using polyhedral oligomeric Vinyl-Silsesquioxanes (POSS) derivatives as crosslinking agents. The results showed improved effects of POSS on thermal stability, mechanical properties, and hardness compared to PDMS polymers prepared with the traditional tetra-functional cross-linkers TMOS and TEOS. The improvements in thermal properties, mechanical properties and hardness could be attributed to the synergistic effect of increasing the dimensionality of the cross-linked networks in new RTV silicone rubbers resulting from the special three-dimensional structure of the new POSS cross-linkers, that are plasticizing self-bonds.

Jitendra Kumar Katiyar and Abdul Samad Mohammed [95], studied the tribological properties of polymer composite coating on silicon wafer. It was concluded that: SU-8/talc/PFPE composite exhibited a hydrophobic surface with higher thermal stability, higher mechanical properties, lower friction coefficient and smaller wear rate, making it a potential candidate to be used in MEMS fabrication. The results of the wetting test showed that the surface of the SU-8 coating changed from hydrophilic to hydrophobic after mixing it with PFPE.

In [96], a new strategy to improve the tribological properties of the polymer by coating with amorphous carbon growing in situ on the surface was realized. The cross-linked structure of the in-situ transition layer served as a barrier to the diffusion of organic polymer chains, followed by the continuous epitaxial growth of the pure aC matrix with continuous carbon plasma treatment. Surface and cross-sectional micrographs showed that the thickness of the in-situ transition layer was about 35nm. The degree of graphitization of aC coatings increased with increasing carbon plasma treatment time. The in-situ transition layer provided strong adhesive strength between the aC coating and the EPC substrates through chemical bonding. The aC segments contributed to the low coefficient of friction and wear rate of EPC substrates with aC surface coating.

Bharadwaja K., *et al.* [97], evaluated the mechanical and tribological performance of epoxy-SiO_2 nano-composites. In order to obtain a high-quality dispersion of epoxy-SiO_2 nano-composites, ultrasonic mixing was used and the results revealed that the bending properties of the epoxy matrix with the introduction of nanoparticles, the hardness and impact resistance, as well as the wear and friction coefficient of the nano-composite were significantly reduced by adding 1% of the volume of SiO_2 nanoparticles to the epoxy matrix. The high mechanical properties of epoxy-SiO_2 nano-composites together with good abrasion, allow the material to normally replace fibres in composites as a partial or full replacement.

The tribological behaviour and mechanical properties of PET and PDMS [98], were evaluated at the nano-scale with respect to environmental conditions. Based on the experimental results, the following conclusions can be drawn: the adhesion force of PET increased near Tg, but showed a similar value at other temperatures. As for PDMS, the adhesion increased steadily with temperature. PET friction was dominated by the deformation mechanism, while PDMS friction is well correlated with adhesion. Despite its softness, PDMS showed no evidence of wear, while significant wear was observed for PET.

A tribological analysis of bulk polymers used in high load applications was carried out in [99]. Four polymers polyetheretherketone (PEEK), polytetrafluoroethylene (PTFE) and aromatic thermosetting polyester (ATSP) were analysed. Researchers have found that high-performance bulk polymers exhibit high tribological properties compared to unfilled polymers. Comparing the four polymers it was concluded that ATSP exhibited better wear resistance while maintaining a moderate COF.

Massimiliano Avalle and Elisa Romanello [100], performed the tribological characterization of modified polymer blends. The results indicated beneficial effects on wear rate. Silicone can be an interesting material for polyurethane and for polyamide, and graphite raised some problems in combination with polyamides. In the case of carbon nanotubes, the harmful influence of polyurethane, CNT caused the formation of residues that prematurely damage the surface.

6. Conclusions

Thermoset resins, as well as thermoplastics, are increasingly used to form composite materials to replace metals. Considerable efforts are being made in all industries to replace metals with composite materials. The main driver of these efforts is low price and improved mechanical properties.

Another benefit of using resins is that the recycled materials can be used to make other objects.

Following the regulations on environmental protection, the management of waste from parts or scrapped vehicles requires that all materials used in engineering be recovered and recycled.

Unfortunately, recycling of thermoset matrix composites is currently limited. Thermoplastic matrix composites offer the potential for recycling or reforming.

Even though epoxy resins are used in a wide range of applications, their poor break strength limits their applicability especially in structural applications.

The hardness of the epoxy could be improved by changing the chemical composition of the resin with a flexible modifier or by reducing the crosslink density.

The toughness of the epoxy resin could be improved, without suffering thermo-mechanical damage, by adding a filler material (rubber, thermoplastic or rigid) as a second phase.

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