Interface Modification and Characterization of PVC Based Composites and Nanocomposites

Veerle Vandeginste and Dharmjeet Madhav

Abstract Weak interfaces in composites result in unsatisfactory stiffness and strength of the composites due to poor stress transfer from the matrix to the fibre. Better interfaces can be achieved by physical and chemical modification techniques. Recent studies show a high interest in incorporation of natural fibres in PVC composites as eco-friendly reinforcing component. Common fillers in PVC composites are calcium carbonate and wood flour. Carbon black, copper and nickel metal powders can be added for conductive applications, and ferrite powder for magnetic applications. Plasma treatment has been applied to wood flour and natural fibres to enhance the interface with PVC matrix. Besides this physical modification method, there are many chemical modification techniques, such as treatment of natural fibres with stearic acid or with sodium hydroxide. The use of coupling agents, such as maleic anhydride, silane, titanate, is also a common treatment method to enhance interfaces in composites. The interface can be characterized by several methods, including Fourier Transform Infrared spectrometry, scanning electron microscopy, X-ray computed tomography, pull-out micromechanical tests, dynamic mechanical analyses and rheological tests.

1 Introduction

Incorporation of a dispersed component into a polyvinyl chloride (PVC) matrix or continuous component can be associated with poor dispersion and poor interfacial adhesion, which results in inadequate properties of the final composites or nanocomposites [1]. For example, in composites with weak interfaces, efficient stress transfer from the matrix to the fibre is compromised, and stress becomes concentrated at the gap between the components which will eventually initiate failure. Moreover, a weak interface or poor dispersion of wood flour in PVC composites have shown to trigger creep behaviour of the composite [2]. Hence, if the bonding between the filler (fibres or particulates) and the polymer matrix is poor, then the intended enhanced

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H. Akhina and T. Sabu (eds.), *Poly(Vinyl Chloride) Based Composites and Nanocomposites*, Engineering Materials, https://doi.org/10.1007/978-3-031-45375-5_3

stiffness and strength or other properties of the composite may not be reached satisfactorily. Therefore, some physical and chemical strategies can be adopted to modify the interface to optimize interfacial adhesion.

PVC is a common thermoplastic material used as matrix in composites with applications in the automotive, housing and construction fields based on the advantageous properties of high mechanical strength and corrosion resistance. A variety of fillers including calcium carbonate, silica, montmorillonite, carbon nanotubes, graphene have been incorporated in PVC composite to improve mechanical and thermal properties, such as elastic modulus, toughness, and heat resistance. Recent review works on PVC composites have focused on: the use of PVC composites as membranes in wastewater treatment, gas separation, pervaporation and electromembrane extraction applications [3]; incorporation of layered double hydroxides as thermal stabilizers in PVC composites [4]; sugarcane bagasse-filled PVC composites [5]; PVC composites with carbon nanofillers [6]; natural fibre reinforced PVC composites [7]; and compressive properties of closed-cell PVC foams [8]. Based on PVC composites studies of the last five years, the recent main interest is on PVC composite membranes for water treatment and electrochemical applications, PVC composites with photocatalytic, radiation shielding or better flame retardancy properties, or PVC composites with wood flour or natural fibres for more eco-friendly and better mechanical properties.

The global attention for natural fibre-based composites is because of the biodegradability, availability, low cost, low density, ease of implementation and good specific properties of natural fibres [9]. The incorporation of natural fibres in thermoplastic composites can increase the elastic modulus of the composite. However, it can decrease the impact strength of the composite [7]; and moisture absorption poses an additional challenge. The incompatibility between the hydrophilic natural fibres (with hydroxyl and other polar groups) and the hydrophobic thermoplastic matrix is the main factor leading to weak interfacial bonding between the fibre and the matrix [10]. Therefore, it is crucial to improve interfacial adhesion between the fillers and the PVC matrix.

The interface between the fillers and the polymer matrix is complex, and it is formed in two stages, first by the contact and wetting process, and second by the formation of the interface during polymer curing, physical and chemical changes. The compatibility between the matrix and fillers can be enhanced by physical and chemical modification, or addition of any compatibilizer or coupling agent [11], and graft polymerization of the polymer [1]. The most common physical treatments make use of plasma or irradiation. Chemical treatments of fillers involves acetylation [12, 13], alkali [14] and benzoylation treatments, and use of coupling agents such as silane [15], aminosilane and titanate. The different interface modification and characterization methods of PVC based composites and nanocomposites will be discussed in this chapter.

2 Fillers Used in PVC Composites

Calcium carbonate and wood flour are commonly used fillers in PVC composites. For conductive applications, carbon black and copper and nickel metal powders may be used, and for magnetic applications, ferrite powder is used. Other fillers involve dolomite, limestone, cellulose, silica, kaolin or montmorillonite clay, talc or calcium sulfate. Papers on PVC composites published in the last five years have a strong focus on the incorporation of natural fibres as eco-friendly reinforcing additives for PVC composites. These studies are on PVC composites with sorghum straw fibres [16–18], rice husk fibres [19, 20], cotton stalk fibres [21–23], palm fibres [24], Eucalyptus fibres [25–29], Citrus Maxima fibres [30], date palm fibres [31], areca sheath fibres [32], bamboo, rice straw, wheat straw and reed straw [33], bamboo fibres and coconut shell particles [34], bagasse fibres [35], corn stalk [36, 37] and coconut fibres [38].

Wood flour or other plant-derived fillers are also common in PVC composites, such as kenaf core powder [39], waste rice husk ash [40], corncob flour [41], wheat husk filler [42], bamboo particles [43], wood flour [44–47], wood flour and amorphous calcium carbonate [48], wood flour and precipitated calcium carbonate [49], olive pit flour and precipitated bio-calcium carbonate for wooden PVC composites [50], raspberry pomace filler [51], and flour of decayed wood [52]. A study on PVC composites with organically modified montmorillonite and pine wood flour suggested the latter can be considered a cheap, eco-friendly and renewable substitute for chalk as filler for PVC based cable insulators with improved mechanical and physical properties [53]. Modified eggshell biofiller has also been suggested as an alternative to conventional $CaCO_3$ from limestone or chalk [54]. Moreover, fly ash can also be used as reinforcement filler [55–59]. Calcium sulphate fibres [60], $CaSO_4$ whiskers [61, 62] and glass fibres [63] are also used in PVC composites for better mechanical properties.

Several fillers are added to improve the thermal stability of PVC composites such as organotin, calcium-zinc and titanium nanoparticles [64], talc, calcined kaolin [65], ground basalt rock microparticles [66], and basalt fibres [67]. Thermal stability and flame retardancy in PVC composites are enhanced by magnesium hydroxide particles, magnesium hydroxide whiskers[68], anhydrous $MgCO_3$ particles [69–72], and magnesium borate hydrate-mechanically activated lignin complexes [73]. Chitosan-modified zinc hydroxystannate and reduced graphene oxide can be used as hybrid flame retardant in flexible PVC composites [74], and chitosan-modified inorganic oxyacid as flame retardant in PVC composites [75].

Radiation protection of PVC composites or shielding against gamma and X-ray radiation is achieved by incorporation of micro-nano-structured PbO and CuO particles [76, 77], Bi_2O_3 particles [78, 79], tungsten microparticles [80], or $Pb(NO_3)_2$ particles [81]. Carbon nanotubes and carbonyl iron has been used for improved microwave absorbing properties of PVC composites [82, 83]. Hexagonal cesium tungsten bronze ($CsxWO_3$) nanoparticles are added in PVC composite films for near-infrared light shielding and high visible light transmission [84]. Solar reflectance and cooling performance of PVC composites is improved with TiO_2 particles [85, 86].

Thermal conductivity is improved in plasticized PVC composites by incorporation of fly ash and carbon black [87, 88], graphite and MgO [89], carbon microspheres [90], graphene [91, 92], multi-walled carbon nanotubes [93] and nanographite [94], reduced graphene oxide [95, 96], graphite and mica [97].

Electrical or electrochemical application studies involve the incorporation of multi-walled carbon nanotubes in PVC composite films with polyaniline for flexible and robust electrodes for high performance supercapacitors [98, 99], carbon nanotubes [100, 101], graphene [102], palladium nanoparticles in blended poly(vinylidene fluoride) and PVC composites [103], graphite with PVC composite as anode for electrochemical degradation of metoprolol [104], multi-walled carbon nanotubes for high water flux PVC composite membranes with effective electrically enhanced antifouling properties [105], functionalized reduced graphene oxide for highly electro-responsive PVC composite gel for advanced artificial muscles and tuneable soft actuator applications [106], carbon black for temperature sensor applications [107], salt-loaded PVC membranes for 3D printed electrodes [108], MoS_2 particles in composite PVC membranes for triboelectric nanogenerators [109], and graphite and chitosan for electrodes [110]. Thermal solar conversion is enhanced with multi-walled carbon nanotubes in PVC composites [111, 112].

Enhanced dielectric properties in PVC composites are obtained with $BaTiO_3$ fillers [113, 114]. Antistatic properties are achieved with multilayer graphene in PVC composite [115]. Nickel-zinc ferrite fillers are used for PVC composites with magnetic properties [116]. PVC composite membranes with magnetic Fe_3O_4/oxidized multiwalled carbon nanotubes composite nanoparticles have also been reported [117]. SiC aggregates are incorporated in PVC composites to increase the surface roughness of the composite and increase the coefficient of friction [118].

PVC composite materials are also used in water treatment applications with Al_2O_3, TiO_2, ZnO and SiO_2 nanoparticles in PVC ultrafiltration membranes [119], diatomaceous earth particles in composites for formaldehyde removal applications [120], Methylene Blue and TiO_2 particles in PVC composite film for photocatalytic technology [121], MnO_2 nanosheets in PVC composites for efficient removal of dye from water [122], and graphene oxide and p-phenylenediamine in PVC composites for heavy metal removal applications [123]. Zinc oxide nanoparticles are used for their antibacterial and antifungal properties [124, 125]. Cu nanoparticles contribute antimicrobial activity and better thermal stability of the PVC composites [126].

3 Interface Modification: Physical Modification Methods

The main types of physical modification methods involve electrical discharge methods such as corona treatment and plasma treatment. Also, ultrasonic irradiation is commonly employed to improve dispersion of fillers, in particular of nanoparticles which tend to otherwise agglomerate. Particles and fibres need to be well dispersed in the polymer matrix to achieve good material properties.

3.1 Plasma Treatment

Plasma treatment is a low cost technology and has a low environmental impact in comparison to the use of chemicals such as coupling agents [127]. Plasma treatment is employed to increase the surface energy through modifying the surface by activation, grafting and etching, which can result in better tensile strength and interfacial shear strength of polymer composites with plasma treated fibres [128–130]. For example, it can increase the surface energy of wood particles by oxidative activation, resulting in the formation of polar functional groups, such as hydroxyl, carboxyl and aldehyde groups [131, 132]. This can then also improve the bonding of these particles with silane coupling agents. The plasma method was used to treat wood particles before chemical modification with tetraethyl orthosilicate (TEOS) to improve the mechanical properties of wood flour PVC composites [46].

3.2 Ultrasonic Irradiation

Ultrasound is an efficient method in polymer chemistry, whereby polymerization reactions can be accelerated, or where higher yields can be obtained under milder conditions [133]. This is due to a unique interaction between matter and energy provided by cavitation-induced sonochemistry [134]. Moreover, ultrasound helps with nanoparticle dispersion and deagglomeration [135]. Mechanical properties of composites are generally improved by using fibres as reinforcement, but small particles can also provide more rigidity of the composite and other properties due to increased contact (surface area) between the filler and the matrix. In the fabrication process of PVC composite films, ultrasonic irradiation was used for dispersion of TiO_2 nanoparticles that were surface treated with vitamin B-1 to help prevent aggregation [136]. Similarly, TiO_2 nanoparticles were treated with bovine serum albumin protein and then incorporated in PVC nanocomposite using ultrasound in both processes [137]. Ultrasonic irradiation was also used in several steps of the preparation of PVC composites with ZnO nanoparticles modified with diacid containing alanine amino acid coupling agent, which led to uniform dispersion of the particles and improvement of the mechanical properties of the nanocomposites [138]. Similarly, ZnO nanoparticles modified with polyvinyl alcohol were dispersed using ultrasonication in the fabrication of PVC nanocomposite films with enhanced mechanical properties [139]. Ultrasonication was also employed in the preparation of melamine terephtaldehyde modified graphene oxide as anticorrosion additive in PVC nanocomposite, which showed a lower change loss percentage of tensile strength in acetone and sodium hypochlorite media [140]. Furthermore, fabrication of PVC nanocomposite with mixed graphene and carbon nanotubes was conducted with ultrasonic irradiation [141].

3.3 Surfactant Modification

A surfactant is a surface active agent, which means that it can reduce the free energy of surfaces and interfaces. Moreover, it self-assembles at interfaces, and forms monolayers at liquid-liquid interfaces and monolayers and aggregates at solid-liquid interfaces. Surfactants are amphiphilic molecules; they have at least one polar head group (hydrophilic) and one nonpolar tail group generally made up of a carbon chain (hydrophobic). In a study on PVC composites with rice straw fibres, graphene oxide nanosheets were modified with surfactant sodium dodecylbenzenesulfonate by adding 0.5 wt% to a solution followed by sonication, and added in the PVC composite to improve the mechanical properties [142].

4 Interface Modification: Chemical Modification Methods

Several types of chemical modification techniques have been applied on a variety of fillers in PVC composites. The focus here is on the main chemical treatment methods that have been applied in PVC composite studies published in the last five years.

4.1 Acid and Acetylation Treatment

Stearic acid is one of the most widely used materials for coating fillers, for example calcium carbonate, to enhance their properties and incorporation in polymer composites. Sorghum straw fibre was pretreated with a mixture of stearic and palmitic acids, which improved the water resistance of the fibre PVC composites [143]. Stearic acid was also used to modify fly ash, using 3 wt% stearic acid in comparison to the fly ash weight [88]. Titanium nanoparticles were modified with folic acid at 5 wt% during ultrasonic radiation at 25 °C (Fig. 1) for incorporation in PVC composites [144]. Acetylation involves the use of a catalyst, generally acetic acid, to remove hydrogen atoms from the hydrophilic hydroxyl groups of cellulose molecules by grafting acetyl groups on those sites [145]. Acetylation improves fibre hydrophobicity. Acetylated fibres have a better resistance against moisture absorption [146] and form strong covalent bonds leading to composites with higher tensile strength and Young's modulus [145]. Palm fibres were treated in an acetic acid solution at 25 °C for 45 min, and then put in an acetic anhydride solution with some drops of sulphuric acid at 50 °C for 2 h [24]. These acetylated fibres resulted in improved mechanical properties of the PVC composites due to a better bonding between the fibre surface and the PVC matrix, and the acetylated fibre PVC composites show the lowest rate of water absorption [24].

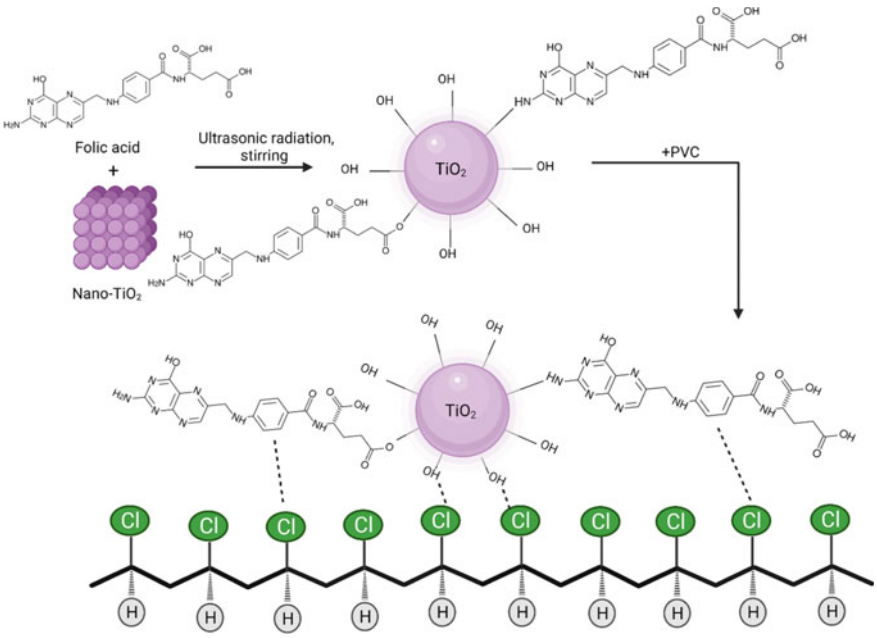

Fig. 1 Mechanism of the modification of TiO_2 nanoparticles with folic acid to incorporate in PVC composites

4.2 Alkaline Treatment or Mercerization

Natural fibres are generally treated with alkaline solutions (often NaOH) to improve the interfacial bonding between the fibre and the polymer matrix, as it can remove amorphous materials such as lignin, pectin and hemicellulose from the surface of cellulose fibre bundles [147]. Moreover, alkaline treatment aids in breaking down the fibres to smaller fibres, a process known as fibre fibrillation, which leads to a higher surface area [148], also favourable for the interfacial adhesion mechanism of mechanical interlocking. Cotton stalk fibres were soaked in a 4 wt% NaOH solution at 90 °C for 2 h under stirring [22]. Palm fibres were treated in a 5 wt% NaOH aqueous solution by immersion at 50 °C for 2 h, and this led to the partial removal of hemicellulose and lignin [24]. Pinewood flour was immersed in 2 wt% NaOH solution for 45 min as alkaline treatment to improve the particle-matrix interactions in PVC composites [53]. Similarly, rice husk filler was put in a 2% NaOH solution at room temperature for 24 h, to remove lignin and pectin and to enhance the compatibility between cellulose of the rice husk filler and the PVC matrix [149]. In another study, rice husk was treated with 5% (w/v) NaOH solution for 30 min at 30 °C, and then kept immersed in distilled water overnight and washed repeatedly before drying in an oven at 70 °C for 12 h [150]. A solution of 10% NaOH was used to treat bagasse fibres for 4 h at 60–70 °C, followed by rinsing with distilled water and drying in oven at 105 °C,

and subsequent bagasse fibre surface functionalization through 2,2,6,6-tetramethyl piperidinyl-1-oxyl (TEMPO)-assisted oxidation of the lignocellulose fibres to form carboxylate groups on the fibre surface [35]. Corn stalk was stirred in an 8 wt% NaOH solution of 80% ethanol and with 1 wt% anthraquinone at 130 °C for 60 min to remove lignin [36]. In addition, in the latter work, a solution of pH 5.5 (by adding sulphuric acid) was used for immersing the corn stalk at 150 °C for 60 min, after the alkaline treatment to remove the hemicellulose component [36].

An alkaline solution can also be used as a pretreatment of inorganic fillers before grafting a coupling agent to improve the effects of grafting. Specifically, ground calcium carbonate powder was mixed with 1.0 mol L^{-1} and 0.1 mol L^{-1} Na_2CO_3 aqueous solution (with 10 wt% solids in the solution) at room temperature and stirred for 2 h to achieve more hydroxyl groups on the filler surface, which leads to more active sites for subsequent reaction with aminopropyltrimethoxysilane (APS) coupling agent [151] (Fig. 2). Similarly, calcium sulfate whiskers were stirred in a 0.03 mol L^{-1} Na_2CO_3 solution at 80 °C for 2 h, washed and dried at 105 °C for 12 h, before modification with silane coupling agent for incorporation in PVC composite [60]. Modification of silver nanoparticles was conducted by dispersion in an ethanol solution with ammonium hydroxide for alkaline treatment, and subsequently tetraethoxysilane was added for silane treatment of the nanoparticles [152]. Fly ash was treated with 6 mol L^{-1} NaOH solution to increase the hydroxyl groups on the surface of the fly ash particles, and subsequently a polyether titanate coupling agent was used for further modification of those particles to improve interfacial adhesion between particles and matrix in PVC composites [59].

4.3 Maleated Coupling Agent

Coupling agents have amphoteric structures, meaning one side of the substance has polar groups with can form strong chemical bonds with for example inorganic fillers, whereas the other side of the coupling agent has nonpolar groups which can form strong interactions with organic polymers. There are many types of coupling agents, and their impact on improved interaction between the polymer matrix and the fillers depends on the composition of those. Moreover, the effect of modification is dominated by the grafting ratio on the surface of fillers. Maleated propylene was employed in several studies on PP composites with wood flour or natural fibres [153–155]. In polylactic acid composites with natural fibre or wood flour, maleic anhydride served as coupling agent for functionalization of polylactic acid to improve compatibility with cellulose fibres [156]. Maleic anhydride was used as coupling agent in wood PVC composites [157], and maleic anhydride grafted on PVC (Fig. 3) was also used as compatilizing agent in olive husk flour PVC composites [42].

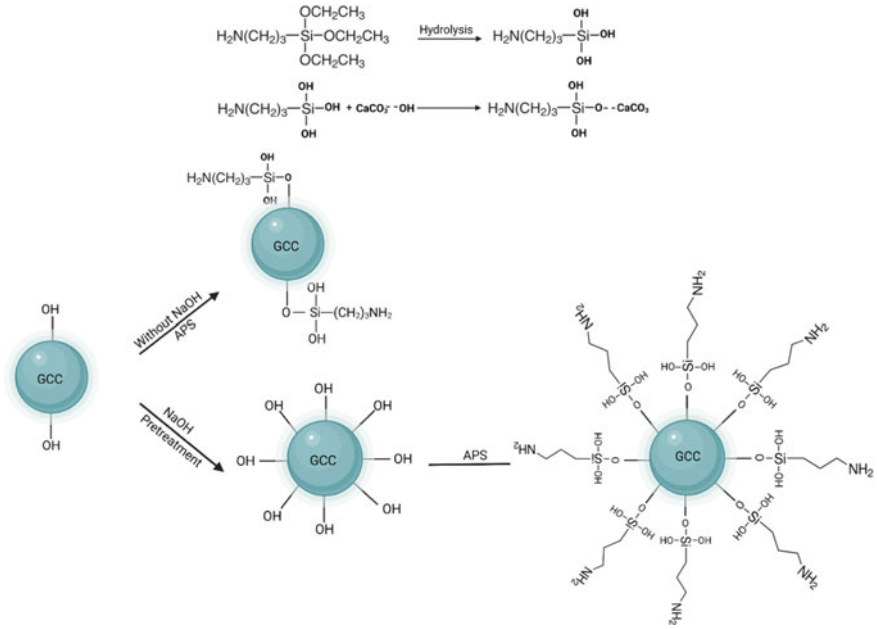

Fig. 2 Procedure of surface modification of ground calcium carbonate (GCC) particles for incorporation in PVC composite

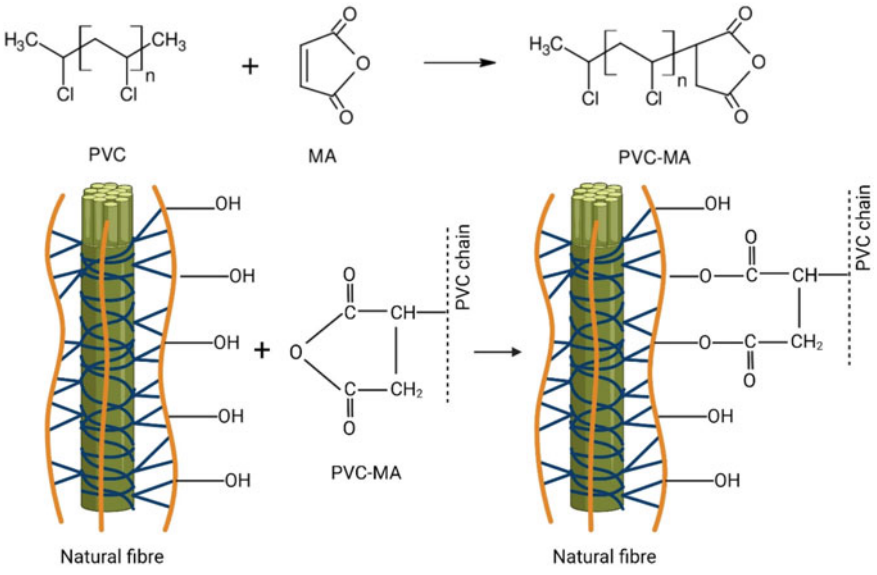

Fig. 3 Grafting of maleic anhydride on PVC to use as coupling agent in PVC composites with natural fibres

4.4 Silane Coupling Agent

The first discovered coupling agent is the silane coupling agent, and it is the most commonly used coupling agent. Silanes consist of a silicon atom which can have up to three reactive groups and one rest group that is generally attached via an alkyl chain. The reactive groups are generally hydroxyl, methoxy, ethoxy or chloride groups. Silane coupling agents can be diluted in either 0.1 wt% acetic acid aqueous solution or a solvent such as methanol, ethanol, propanol or benzene, or a mixture of water and ethanol. The generally recommended dosage of silane coupling agent is 0.8 to 1.5% of the material being processed. Silane is used as a coupling agent because it can interact both with organic polymer matrix and with, for example, inorganic oxide filler, and it aims to reduce stress at the matrix-filler interface and prevents the filler from being affected by moisture.

Zinc oxide nanoparticles were functionalized with polydimethylsiloxane by ultrasonic and constant temperature method for incorporation in plasticised [125] and rigid PVC composites [124]. Gamma-aminopropyltrimethyoxysilane (also called KH-550 or 3-aminopropyltriethoxysilane) was used as a compatibilizer for waste rice husk ash PVC composites [40]. Similarly, this coupling agent was used to chemically treat rice husk fibres (at 2 wt% of rice husk, and diluted with an 80/20 methanol/distilled water mixture with pH adjusted to 4 by using acetic acid) by soaking for 2 h and then drying, to achieve better mechanical properties of PVC composites [158]. The 3-aminopropyltriethoxysilane coupling agent (KH-550) was also used to treat sorghum straw fibre (at 3 wt% compared to fibre, dissolved in ethanol at a volume ratio of 1–5) by spraying and subsequent drying for incorporation in PVC composite [18]. Moreover, fly ash was chemically treated with this coupling agent in recycled PVC composite [56]. Ground calcium carbonate particles were modified with this coupling agent (at 1.5 wt% compared to ground calcium carbonate) in an aqueous solution at 60 °C and stirred for 2 h, to enhance the interfacial bonding with the PVC matrix [151]. Silane coupling agent KH-550 was used in PVC composites with chitosan-modified zinc hydroxystannate flame retardants whereby zinc ions are replaced by chitosan cations [74]. The interaction of this coupling agent with inorganic filler is depicted in Fig. 4.

The effect of KH-550 and methylene diphenyl diisocyanate (MDI) coupling agents were compared in wood veneer PVC (and other thermoplastic) composites and showed that the interfacial bonding was higher using these coupling agents in PVC composites compared to LDPE, HDPE or PP composites [159]. Moreover, the MDI coupling agent improved the interfacial bond strength between wood and thermoplastic and the mechanical properties more than the KH-550 silane coupling agent [159]. Calcium sulfate whiskers were modified with silane coupling agent by stirring in a solution with 90 wt% alcohol and 1 wt% 3-aminopropylsilane at 60 °C for 4 h [60, 61]. Diatomite was modified with gamma-methacryloxypropyltrimethoxysilane coupling agent to introduce hydrophobic chains and improve compatibility of this modified diatomite and PVC matrix [160]. Bismuth oxychloride (BiOCl) nanosheets were modified with methacryl-functional silane coupling agent (KH-570) at 2 wt%

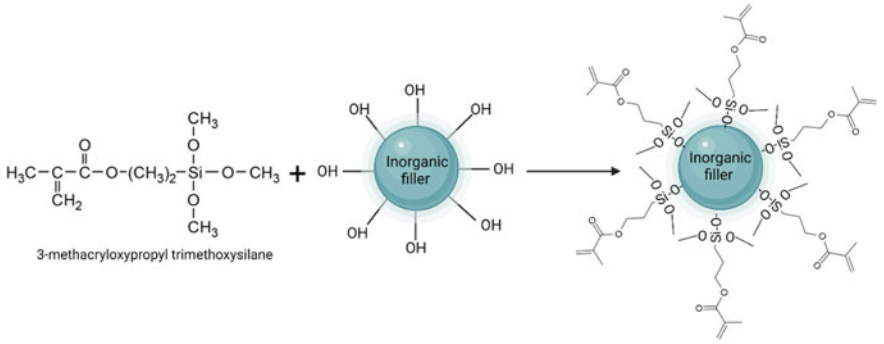

Fig. 4 Surface modification of inorganic filler with KH-550 coupling agent

compared to BiOCl in 95% ethanol solution by stirring and then drying under vacuum, and they were then used a green inorganic flame retardant in flexible PVC [161]. Hydrous manganese dioxide nanoparticles were functionalized with 3-mercaptopropyl trimethoxysilane coupling agent [162]. Furthermore, tetraethylorthosilicate (TEOS) is mainly used as crosslinking agent in silicone polymers, and it is also employed for surface modification, for example, to modify the surface of halloysite nanotubes in reed flour PVC composites [163].

4.5 Titanate Coupling Agent

Besides silane coupling agents, also aluminate, borate, rare earth and titanate coupling agents are used to improve bonding at the interface. There are more than 70 titanate coupling agents, and they are generally divided into four types, namely chelating, coordination, pyrophosphate and single alkoxy types. The choice of titanate coupling agent is related to the type of filler; they are suitable for calcium carbonate, titanium dioxide, barium sulfate, kaolin, talc, mica, but not really suitable for aluminium oxide and glass fibre. The general dosage of titanate coupling agent used is 0.1–2.0% of the material being processed. Using titanate coupling agent to modify inorganic fillers (Fig. 5) can help improve impact strength, mechanical properties of the composite, dispersion of the fillers and reduce the viscosity of the system. Commercial titanate coupling agents and a new polyether titanate coupling agent were used to modify calcium sulfate whiskers for incorporation in PVC composites for better thermal stability [61]. The latter titanate coupling agent was also used for surface modification of magnesium hydroxide particles and magnesium hydroxide whiskers for incorporation in PVC composites for better flame retardancy properties [68]. Moreover, carbon microspheres were modified with this polyether titanate coupling agent in PVC composite [90], and also basic magnesium carbonate was modified with this coupling agent [71]. Butyl titanate coupling agent was used to modify nanosized titanium dioxide to synthesize PVC composites [64], and isopropoxy trioleate acyl

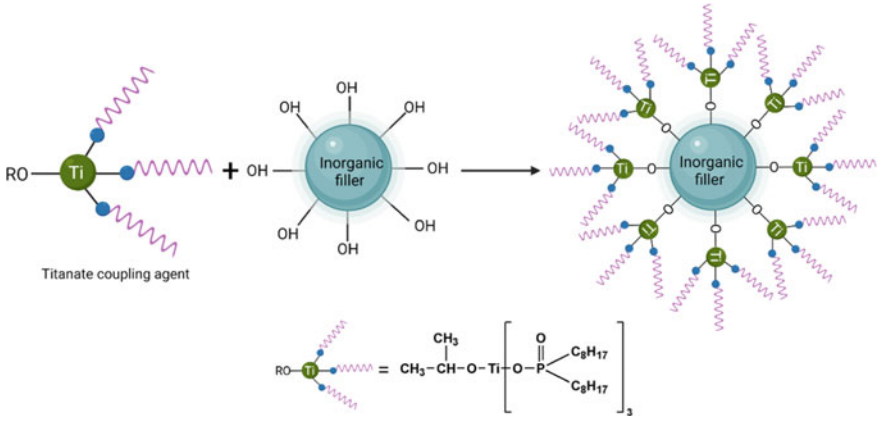

Fig. 5 Surface modification of inorganic filler with titanate coupling agent

titanate (KTTO) coupling agent was employed to chemically modify the surface of silica microparticles and improve the interfacial adhesion PVC composites [164].

4.6 Benzoyl Chloride Treatment

Benzoylation treatment of natural fibres involves the substitution of hydroxyl groups of cellulose for benzoyl groups (Fig. 6). This chemical treatment improves fibre-matrix adhesion, resulting in composites with higher tensile strength and toughness, better thermal stability and reduced water absorption [165]. Kenaf core powder was treated/grafted with benzoyl chloride in PVC composites [39]. Olive husk flour was also chemically modified through benzoylation, by first soaking olive husk flour in 18% NaOH solution for 30 min, then washing, before being stirred in a 10% NaOH solution with benzoyl chloride at room temperature for 15 min [11]. The benzoylation treatment led to higher Young's modulus and reduced moisture absorption rate of the PVC composite [11].

4.7 Chitosan-Based Treatment

Chitosan is an interesting biopolymer due to its biocompatibility, non-toxicity and good mechanical properties. Chitosan can be produced from chitin and it has many hydroxyl and amino functional groups. The amino groups of chitosan can be modified to form hydrophilic structures that can strengthen bonding with inorganic particles and polymers [166]. A cinnamaldehyde-chitosan derivative was synthesized as

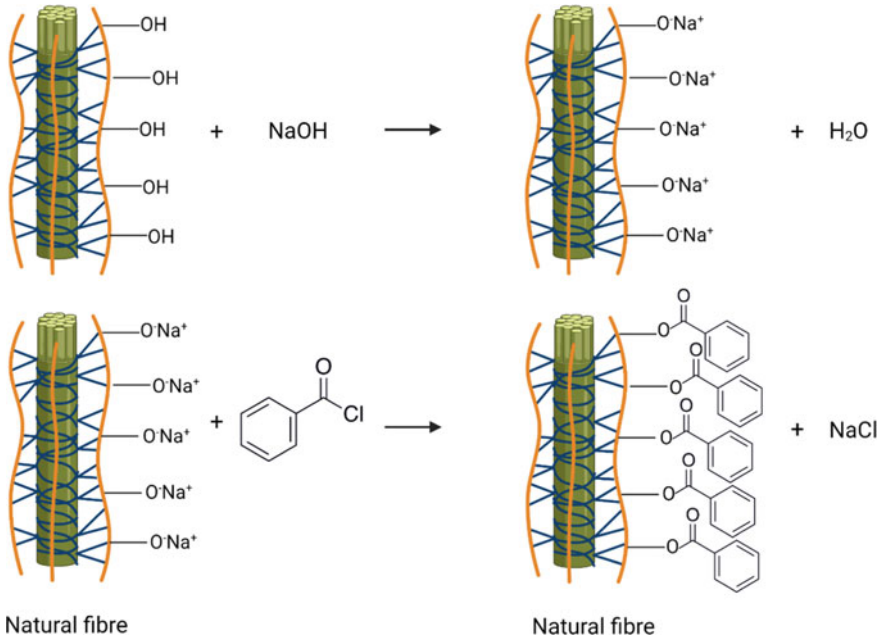

Fig. 6 Benzoylation of natural fibres by treatment with NaOH and benzoyl chloride solutions

a biopolymer coupling agent to modify carbon microspheres and improve compatibility between the latter and PVC composite [90]. Chitosan was coated on the surface of magnesium hydroxide sulfate hydrate whiskers, by stirring them in a mixture of 1% acetic acid aqueous solution with chitosan (of up to 0.5%) at room temperature for 2 h, for a better adhesion and compatibility with PVC matrix [167]. The mechanism of chitosan coating on inorganic filler is presented in Fig. 7.

4.8 Phthalate Treatment

Dioctyl phthalate is generally used as a plasticizer in the production of flexible PVC. It has also been employed to modify the surface of antimony trioxide nanoparticles (Fig. 8) using high energy ball milling to improve dispersion of the particles and compatibility with PVC matrix to obtain PVC composites with good mechanical and flame retardant properties [168].

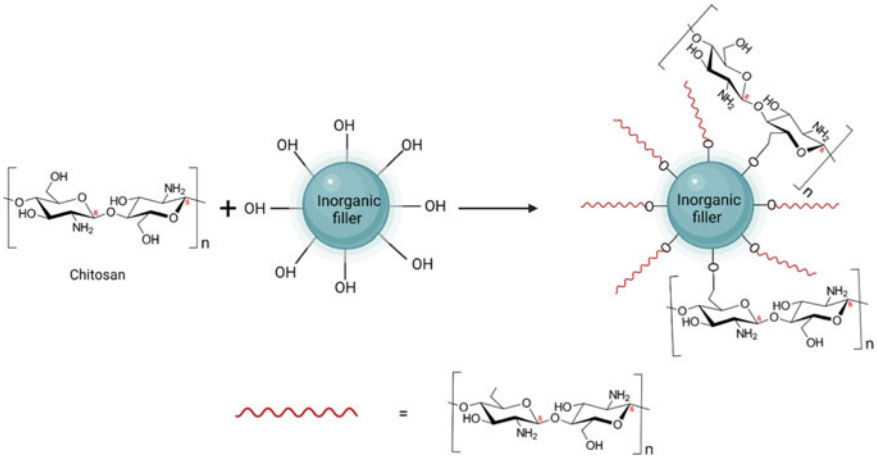

Fig. 7 Surface treatment of inorganic filler by coating with chitosan in slightly acidic aqueous solution

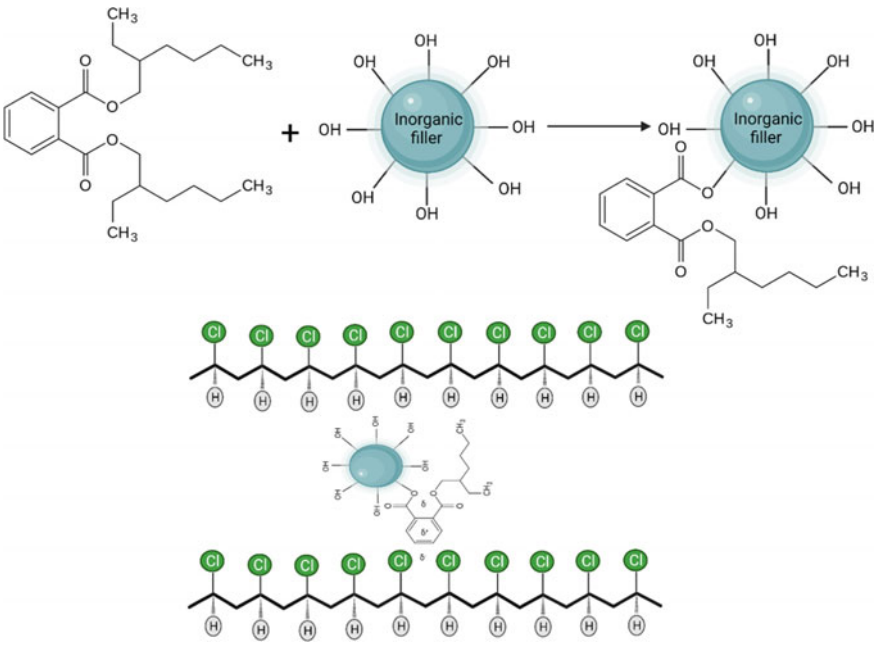

Fig. 8 Chemical modification of the surface of inorganic filler using phthalate compound

5 Interface Characterization Methods

The mechanical properties of composites that are commonly tested, such as tensile strength, tensile modulus, elongation at break, impact strength, give information on the reinforcing effect of the fillers in the composites. These properties are influenced by the adhesion strength between the filler and the matrix of the composite, and thus the measurements of these properties at the macro-level give indirectly an estimate of the performance at the interface. Still, here the focus lies more specifically on the interface characterization methods.

5.1 Fourier Transform InfraRed Spectrometry

Identification of functional groups in a material as well as the interactions of components in composites can be obtained by Fourier Transform InfraRed (FTIR) spectrometry. This method is applied to verify successful modification of fillers. For example, bonding enhancement of wood flour in PVC composite by adding natural chitosan was investigated with FTIR spectra [169]. A higher amount of chitosan added in the composite led to a higher transmittance of the C-Cl bond peaks and very slight decrease in wavenumber, meaning the C-Cl bonding was weaker and that both C and Cl had stronger interactions with other ambient atoms; similarly, the transmittance of amino and hydroxyl groups decreased, meaning higher bonding forces of chitosan [169]. The hydrolysis effect of alkali treatment on PET fibres was confirmed by the decrease in intensity of hydroxyl peaks in absorbance FTIR spectra [170]. The peak of olive pit flour for O-H stretching was shifted towards higher wave number upon addition of PVC, which was interpreted as the formation of H bonds among hydroxyl groups of cellulose or lignin in olive pit flour and hydrogen in PVC, confirming the interaction between olive pit four particles and PVC matrix [106]. Figure 9 shows FTIR spectra of the authors' research on PVC composite membranes with 8 wt% precipitated $CaCO_3$ microparticles of mussel shell $CaCO_3$ microparticles. The PVC composites were made with untreated microparticles and with stearic acid treated microparticles. The stearic acid treatment was conducted in two different ways: (i) in ethanol at room temperature, and (ii) in water at 45 °C. The stearic acid modification leads to a change in the transmittance of the peaks, and very slight shift in wavenumber.

5.2 Scanning Electron Microscopy and Energy Dispersive Analysis of X-rays

Scanning electron microscopy (SEM) can be used to study the morphology of the interface between fillers and the PVC matrix. Micro-scale damage characteristics

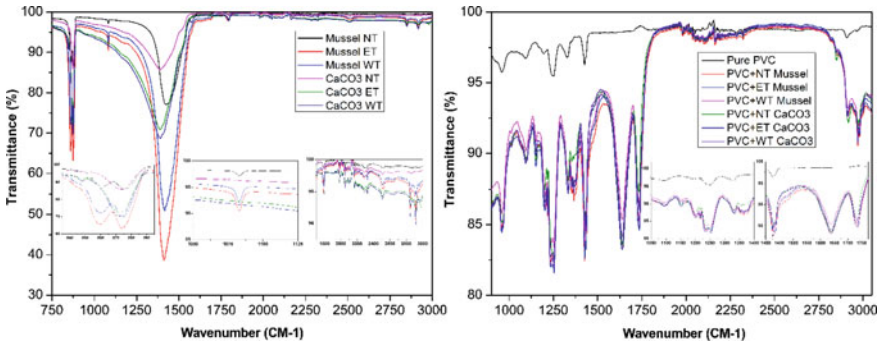

Fig. 9 FTIR spectra of mussel shell $CaCO_3$ microparticles and precipitated $CaCO_3$ microparticles that are untreated (NT), treated with stearic acid in ethanol (ET) or treated with stearic acid in water (WT); and FTIR spectra of pure PVC and of the PVC composites with the six types of (un)treated microparticles

from tensile tests can be investigated and identified using SEM imaging. For example, shear failure, matrix cracking, fibre breakage, fibre fracture and fibre pull-out were observed in PVC composites with untreated cotton fibres, based on SEM imaging [23]. Moreover, the dispersion of fillers in the polymer matrix can be evaluated. SEM images of PVC composites with $CaCO_3$ nanoparticles reveal that the nanoparticles treated with titanate coupling agent were better dispersed and showed better interfacial adhesion in comparison with nanoparticles that were treated with sodium stearate [171]. SEM pictures enable identification of smooth and clean surfaces due to pulling out of bagasse fibres from PVC matrix in fractured composites, whereas the surfaces are a bit rough in fractured PVC composites that contain bagasse fibres treated with benzoic acid [172]. Moreover, the untreated fibre PVC composite showed larger empty spaces due to the aggregation of fibres, in contrast to the observations in the benzoic acid treated fibre PVC composite [172]. SEM images show that addition of finer chitosan particles, with thus a larger specific surface, leads to a more effective bond at the interface of the wood fibre and PVC matrix [169]. In PVC composites with untreated calcium sulfate whiskers, SEM images demonstrated whisker pull-outs and gaps between whiskers and matrix, whereas whiskers treated with glutaraldehyde cross-linked polyvinyl alcohol were attached to the PVC matrix by a rough adhesive interface and the whiskers were well dispersed in the matrix [173]. In PVC composites with calcium sulfate whiskers that were treated with KH-550, titanate coupling agent or stearic acid, the interactions between the whiskers and the matrix seemed enhanced in comparison to untreated whiskers, but still some voids and gaps were observed in the composites [173]. The effect of alkali treatment of PET fibres, and subsequent coating with SiO_2/tributyl citrate hybrid sizing agent, was investigated with SEM, demonstrating the pits and grooves on the PET surface after alkali treatment and the deposition of nanoparticles scattered on the fibre surface upon treatment with the sizing agent, with both treatments increasing the specific surface area [170]. The surface of untreated glass fibres pulled out of PVC composites investigated with

SEM were very smooth, whereas the glass fibres treated with coupling agent had some PVC attached to them, with the effect being stronger with KH-550 coupling agent than KH-660 and KH-570 coupling agents, which was also confirmed by the mechanical property tests of tensile strength, flexural strength, impact strength and interlaminar shear strength [63]. Improved particle dispersion and enhanced interfacial adhesion was detected by SEM in olive pit flour PVC composites with loading of up to 10% precipitated bio-calcium carbonate particles, after which the properties deteriorated [106]. SEM imaging of PVC composite thin films with hybrid ceramic filler was also used to demonstrate effective hybridization between V_2C and Cu_2O phases with agglomeration of fine Cu_2O particles at the surface of V_2C phase due to the higher surface activity and the creation of a rough surface [174].

Also in the authors' research on PVC composites with 8 wt% nontreated $CaCO_3$ microparticles and stearic acid treated $CaCO_3$ microparticles, SEM images showed better dispersion of the stearic acid treated particles than the untreated ones in the PVC composite membrane. Energy dispersive X-ray (EDX) analysis was performed to map the distribution of the microparticles in the polymer composite, further confirming and visually presenting the better dispersion of microparticles in the composites with stearic acid treated particles (Fig. 10). With the increasing attention towards incorporation of renewable fillers or food waste, e.g. egg shell waste $CaCO_3$ fillers [175], the incorporation of mussel shell $CaCO_3$ microparticles (8 wt%) in PVC composite membrane was also tested, and an improvement in the distribution of the stearic acid treated particles was observed with less agglomeration in comparison to untreated mussel shell waste $CaCO_3$ microparticles in PVC composite membrane (Fig. 11).

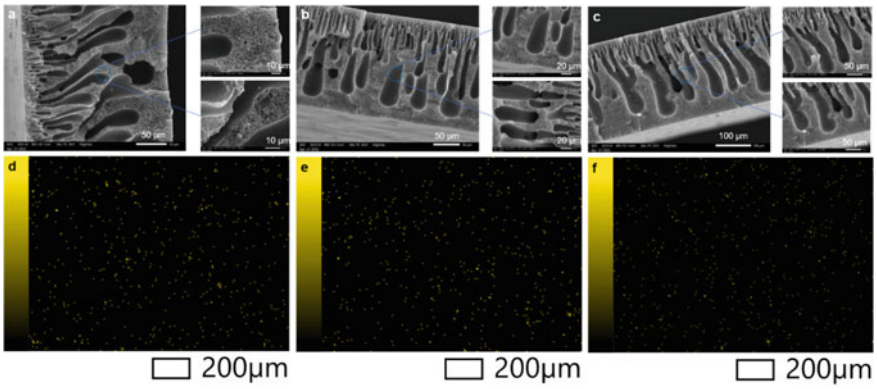

Fig. 10 SEM images of $CaCO_3$ PVC composite membranes with **a** untreated $CaCO_3$ microparticles, **b** $CaCO_3$ particles treated with stearic acid in ethanol at room temperature, **c** $CaCO_3$ particles treated with stearic acid in water at 45 °C, and EDX images of Ca distribution in $CaCO_3$ PVC composite membranes with **d** untreated $CaCO_3$ microparticles, **e** $CaCO_3$ particles treated with stearic acid in ethanol at room temperature, **f** $CaCO_3$ particles treated with stearic acid in water at 45 °C

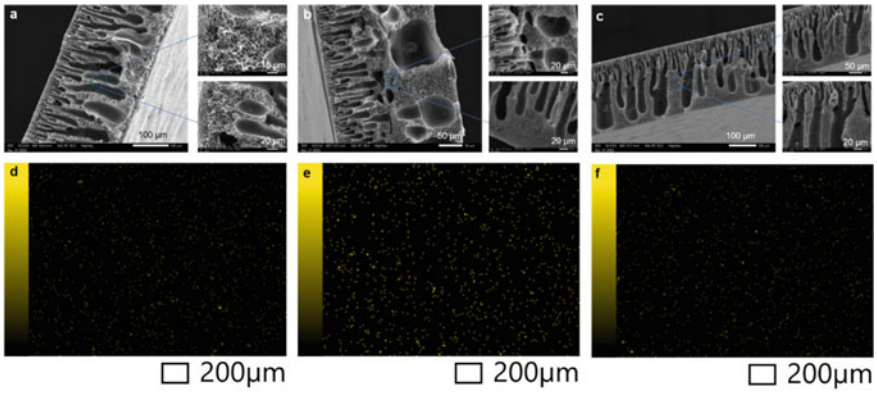

Fig. 11 SEM images of mussel shell waste $CaCO_3$ PVC composite membranes with **a** untreated mussel shell waste $CaCO_3$ microparticles, **b** mussel shell waste $CaCO_3$ particles treated with stearic acid in ethanol at room temperature, **c** mussel shell waste $CaCO_3$ particles treated with stearic acid in water at 45 °C, and EDX images of Ca distribution in mussel shell waste $CaCO_3$ PVC composite membranes with **d** untreated mussel shell waste $CaCO_3$ microparticles, **e** mussel shell waste $CaCO_3$ particles treated with stearic acid in ethanol at room temperature, **f** mussel shell waste $CaCO_3$ particles treated with stearic acid in water at 45 °C

5.3 X-ray Computed Tomography

The 3D structure of polymer composites can be imaged using X-ray computed tomography (XCT). It is a non-destructive technique that can generate a 3D image based on a stack of X-ray projections taken from several angles. The advancements in this technique over the last decade have enabled very high spatial resolution, in the form of nanotomography. Thus, this method allows the reconstruction of filler 3D distribution in the polymer matrix, filler percentage, orientation and dimensions of the fillers, and porosity. Moreover, at high resolution, the interface between fillers and polymer matrix can be investigated in 3D. Also, the effect of coupling agents or other interface modification techniques can be studied, for example, in terms of filler distribution. Imaging using XCT was used in the analysis and interpretation of shear fatigue of balsa wood and PVC foam sandwich core composites, whereby XCT analysis enabled quantification of the thickness of resin absorbed by foam core [176]. The XCT method was also applied to investigate deformation-induced dilation of PVC composites with mineral fillers, and different relative density distributions were identified for different geometries of the specimens that were subjected to tensile tests [177].

5.4 Pull-Out Micromechanical Test

A micromechanical analysis of the interfacial adhesion between a fibre and polymer matrix can be done using a pull-out test. This type of test has been commonly used on glass fibre reinforced composites, and is more recently also adopted for composites with natural fibres. The technique involves pulling a fibre out of a polymer block, small cylinder or micro-droplet, which is held by a microvise. A pulling force is then applied in the axial direction, which induces mainly tensile stress in the fibre and also shear stress at the interface of the fibre and the matrix, by increasing the displacement at constant rate while the force is monitored until the fibre breaks or is pulled out. Data generated with this test allow the calculation of the apparent interfacial shear strength. Although the pull-out micromechanical test is a common method to evaluate interfacial adhesion of fibres (and the effect of interface modification) in polymer matrices, the authors did not encounter studies focused on this technique in PVC composites.

5.5 Dynamic Mechanical (Thermal) Analysis

Dynamic mechanical analysis is used to study the viscoelastic response of polymers or polymer composites. This method is also applied to evaluate the interfacial interaction between particles and polymer matrix in composites. Generally, the motion of polymer chains is difficult near the interface, when there is a strong interfacial adhesion in the composite. Studies have reported that the storage modulus and loss modulus is much higher for PVC composites with calcium sulfate whiskers treated with titanium coupling agent than those with untreated whiskers, showing the efficient improvement of interfacial interaction, restricting deformation of the matrix [178]. A study on PVC composites with multi-layered graphene shows that the addition of graphene results in composites with a higher storage modulus than that of pure PVC, confirming the increase in tensile modulus or stiffness in static mode [179]. The composites have also a higher loss modulus and loss factor than pure PVC due to higher energy dissipation and higher damping of the composites under cyclic deformation conditions, which is interpreted by the larger interfacial area and interfacial friction-sliding between PVC chains and multi-layered graphene [179]. A lower value for the mechanical loss factor is measured in PVC composites with diatomite powder in comparison to pure PVC due to a restriction in the motion of molecular chains [160]. The mechanical loss factor in wood flour PVC composites showed a similar trend for pristine and recycled composites, and some recycled composites samples indicated a higher mechanical loss factor [45]. These higher values of the recycled composites suggest more viscous than elastic behaviour in comparison to the pristine composites. Moreover, the peak of the dynamic mechanical thermal analysis spectra showed a lower glass transition temperature of the recycled composites, which can be

linked to the reduced fibre aspect ratio due to reprocessing, leading to easier polymer molecular motions, and thus reduced glass transition temperature [45].

5.6 Rheological Properties

The rheological behaviour of a polymer composite with fillers is affected by the affinity of the filler and the polymer. Adhesion of nanoparticles to polymer matrix leads usually to a higher viscosity of polymer composites, and the melt viscosity reaches a stable value at high shear stress range. A higher melt viscosity was also observed for PVC composites with $CaCO_3$ nanoparticles than for pure PVC [180]. Moreover, the melt viscosity of PVC composites with treated $CaCO_3$ nanoparticles was lower than that of PVC composites with untreated $CaCO_3$ nanoparticles, interpreted to be caused by the better dispersion of treated $CaCO_3$ nanoparticles in the PVC matrix and lower interaction between the nanoparticles [180].

6 Conclusion

Good adhesion between fillers and the polymer matrix is very important to reach the targeted performance and properties of the polymer composite. Conventionally, fillers are added to polymer composites to improve the mechanical properties, with a recent interest in the use of natural fibres as reinforcement. Moreover, other properties of PVC composites, such as radiation shielding and conductive properties have received more interest recently. In particular, PVC membranes for water treatment and electrochemical applications have attracted more attention in the last years. Physical and chemical modification methods can be applied to enhance interfacial adhesion in the composite material. This chapter has focused on plasma treatment, ultrasonic irradiation and surfactant modification as physical interface modification methods. In terms of chemical modification methods, acid, acetylation and alkaline treatment, the use of different coupling agents (maleated, silane, titanate), benzoyl chloride, chitosan-based, and phthalate treatment have been discussed. Furthermore, several interface characterization methods that have been used in research on PVC composites have been reviewed, with particular focus on the effect of interface physical and chemical modification methods, and Fourier transform infrared spectroscopy, scanning electron microscopy – energy dispersive X-ray analysis, X-ray computed tomography, dynamic mechanical analysis and rheological methods are reported.

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