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Clay-Containing Polysulfone Nanocomposites

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1. Introduction

Although there are several definitions of composite materials (Callister, 1994; Chawla, 1987; Hull, 1981; Padilha, 1997; Van Vlack, 1984), composites are artificial multiphase materials in which their constituents are chemically distinct and separated by an easily distinct interface (Moffatt et al., 1964).

Some familiar materials are examples of composites: the concrete used in buildings, car tires, the asphalt of our streets. The distinction of artificial materials is necessary due to materials that could be included in this definition, but which occur in a natural way, as, for example, wood (lignine + cellulose fibers), bones (collagen + apathite) in which the combination of different phases results in an enhanced material as compared to the individual phases.

Polymer/ clay nanocomposites are a new class of composites with polymer matrixes in which the dispersed phase is the silicate constituted by particles that have at least one of the dimensions in nanometer level. One of the components is the matrix, in which the particles of the second material are dispersed. The most used mineral particles in these nanocomposites are smectitic clays (montmorillonite, saponite and hectorite), having their particles lamellae morphology with sides in micrometer level and thickness around one nanometer (Alexandre & Dubois, 2000; Esteves et al., 2004).

The montmorillonite particles are extremely small and, therefore, their structure can only be studied by X-ray diffraction. The unitary layer of the montmorillonite consists in three sheets: one octahedral sheet composed of hydrargilite-brucite included between two tetrahedral sheets composed of silicon and oxygen. These sheets are combined in such a way that the tetrahedral edges of each silica layer form a common layer with one of the hydroxyl layers of the octahedral sheet, with oxygen atoms instead of hydroxyl groups (Papin, 1993). The layers are continuous in the a and b directions and are stacked in the c direction (Fig. 1). Water and other polar molecules can enter the unitary layers causing an expansion in the c direction. The dimension of the c axis of the montmorillonite varies from 9.6 Å, when any polar molecule is between the unitary layers, until complete separation in some cases (Brigatti et al., 2006).

The interest in the production of polymer nanocomposites comes from the large range of possible applications which involves this type of material. Features such as higher resistances towards high temperatures, UV radiation, high gas impermeability, low expansibility and processing flexibility can be achieved by the production of this type of nanocomposite. Moreover, polymers have a great well-established variety of types of processing as, for example, blending, compression, fusion, mixture and polymerization,

among others, satisfying premises for the production of the most different types of products, such as films, membranes, automotive parts, electronics, packaging materials, etc (Ke & Strove, 2005).

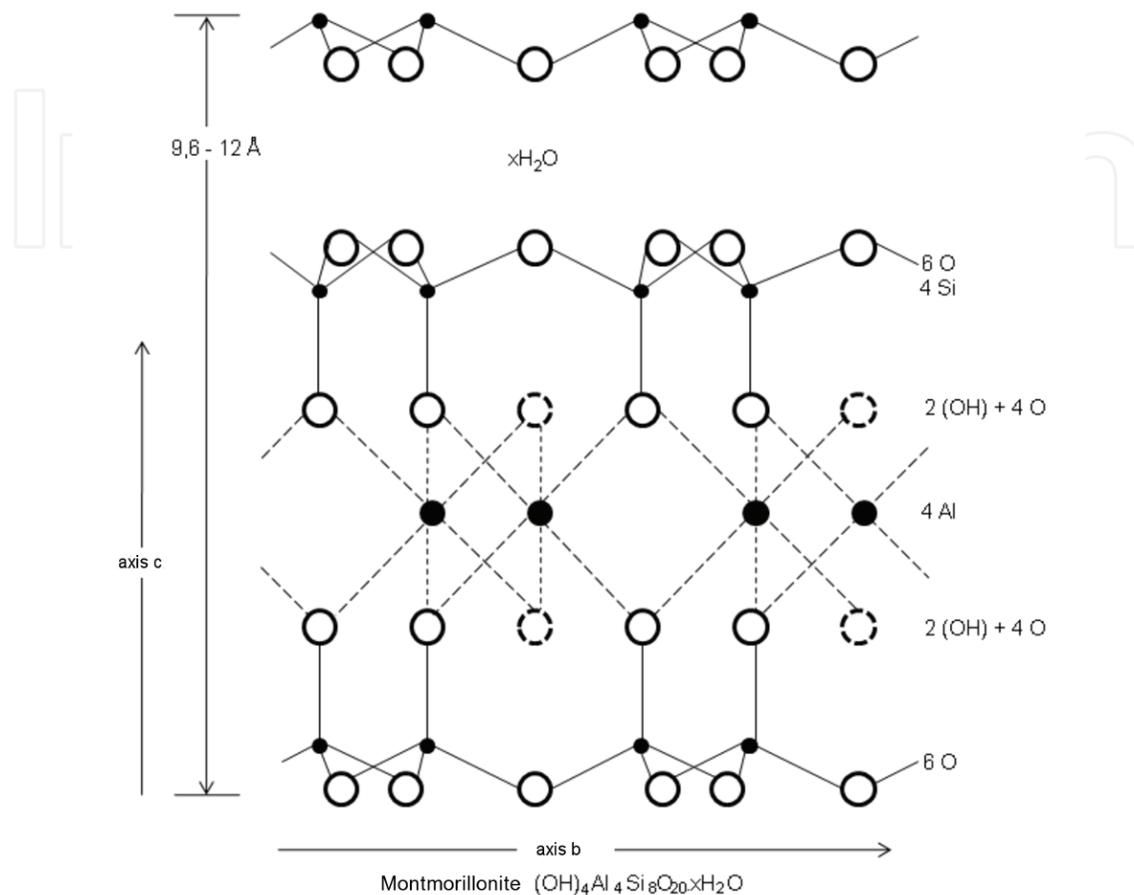


Fig. 1. Schematical representation of the montmorillonite structure (Papin, 1993).

One example of a polymer that has a wide versatility is polysulfone. Its importance is due to its mechanical, thermal and chemical stability as well as its excellent processability. A field which largely uses polysulfone is the membrane area in applications such as microfiltration, ultrafiltration, reverse osmosis and gas separation (Summers et al., 2003). However, the further enhancement in its physical property of polymer substrate is desirable for specific and enhanced applications.

Several reviews about polymer-clay nanocomposites were carried out in the last years (Choudalakis & Gotsis, 2009; Mittal, 2009; Pavlidou & Papaspyrides, 2008; Ray & Okamoto, 2003; Schmidt et al., 2002; Yeh & Chang, 2008). Considering the great attention that has been paid to polymer-clay nanocomposites and to polysulfone, presenting researches which involve polysulfone-clay nanocomposites is opportune. In this chapter, a brief history of thermoplastic nanocomposites, nanocomposite morphologies, types of polymer-clay nanocomposite production, modifications in polymer and clay structures which allow them to be used in nanocomposite preparation will be presented. Hence, having these concepts developed, works related to the preparation and characterization of clay-containing polysulfone nanocomposites, giving priority to papers and patents which describe this material, will be detailed.

2. The history of thermoplastic nanocomposites

Until the early 1970s, minerals were used in polymers only as fillers, aiming to reduce costs, since these fillers are generally heavier and cheaper than the polymer to which they are added.

During the 1970s, some changes occurred in the international scenario, as, for example, the vertiginous increase in the petroleum price during and after the 1973 and 1979 crisis (Rabello, 2000). These facts, together with the introduction of polypropylene in commercial scale, besides the development of materials with mica, glass spheres and fibers, talc and calcium carbonate, led to an expansion of the ceramic raw material market as fillers and to the beginning of the research into the interactions between polymers and fillers.

During the 1980s and 90s, these investigations advanced, leading to a higher understanding of the mechanisms of interactions with different types of polymers. All these researches raised mineral from the simple filler condition to the functional filler condition.

Although polymer-clay nanocomposites appeared before the 1980s (Carter et al., 1950), only at the end of this decade, works which guided to the development of thermoplastic nanocomposites began to appear in the literature, which were developed by Toyota by using polyamide 6 and organophilic clays specially prepared for this nanocomposite (Kawasumi, 1989; Okada et al., 1988).

These works guided to the first commercial applications of these nanocomposites in timing belt covers of Toyota vehicles, in collaboration with UBE company, a Japanese polyamide 6 industry. This material had only 5 wt.% special clay which sensibly improved the features of this material as compared to that of pure polyamide 6. According to Toyota, the nanocomposite formation provided an increase of 40% in the rupture tension, 60% of the tensile modulus and 126% in the flexion modulus, together with the increase in the thermal distortion temperature from 65 to 152°C in comparison with pure polymer.

From then on, several other companies have presented thermoplastic nanocomposites, the great majority of them with polyamide and polypropylene for automotive applications. The choice for the automotive sector is justified by the need of obtaining sufficient volume of this material, which is a great problem nowadays. Still, General Motors, the industry that uses the most nanocomposites in the world, used only 245 tons of these materials in 2003 (Cox et al., 2004; Patterson, 2004).

Another application that has gained attention is the gas barrier, using polyamide and polyester as the matrix (Conway, 2004; Goldman & Copsey, 2004). However, a large-scale commercial application of these materials is still necessary, since, although their performance is better, the competition with other materials is harmed when the cost-benefit ratio is analyzed.

3. Polymer-clay nanocomposite morphologies

Depending on the interfacial interactions forces between polymer matrix and silicate, three different types of nanocomposites are thermodynamically accepted (Fig. 2):

- intercalated nanocomposites: in the intercalated nanocomposites, the insertion of polymer matrix in the silicate structure occurs in a regular way, crystallographically, by alternating polymer chains and silicate layer, whose distance between each other varies from 1 to 4 nm, distance in the range of the polymer chain length;

- flocculated nanocomposites: they are the same as intercalated nanocomposites, except for the fact that some silicate layers are, sometimes, flocculated due to the interactions between hydroxyl groups of the silicate;
- exfoliated nanocomposites: in the exfoliated nanocomposites, the individual clay mineral layers are randomly separated in a continuous polymer matrix by an average distance which depends on the clay charge. Generally, the clay content in an exfoliated nanocomposite is much lower than in an intercalated nanocomposite (Ke & Stroeve, 2005; Ray & Okamoto, 2003).

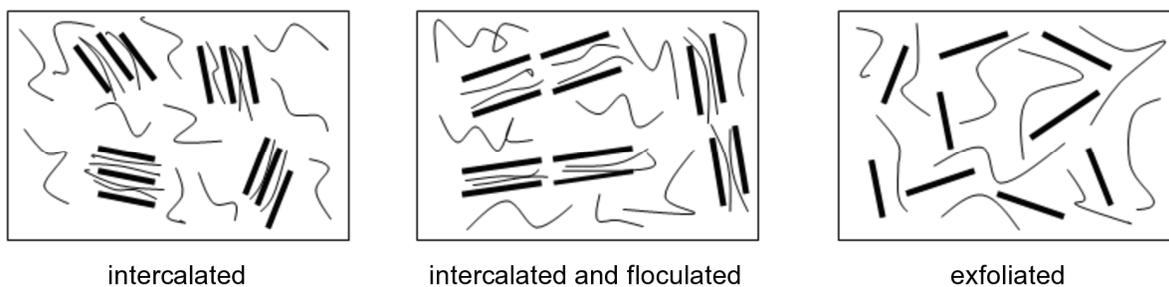


Fig. 2. Illustration of the three types of thermodynamically accepted nanocomposites (Anadão et al., 2011)

4. Nanocomposite production methods

Four methods are used in nanocomposite preparation. In situ polymerization was the first method developed. In this technique, a monomer is used as a medium for the clay dispersion and favorable conditions are performed in order to carry out the polymerization between the clay mineral layers. Polymerization can be initiated by heat or radiation, by the diffusion of an adequate initiator or a fixed catalyzer inside the layers before the filling step by the monomer.

There are cases in which complete exfoliation occurs, since the driving force for complete exfoliation is polymerization. Due to the fact that the clay mineral has high surface energy, the clay mineral layers exert attraction on the monomer units towards the inside of the galleries until equilibrium is reached. The polymerization reactions occur between the layers with lower polarities and displace the equilibrium, aiming at the diffusion of new polar specimens between the layers to progressively exfoliate the clay mineral.

The second method is the solution dispersion in which the silicate is exfoliated in single layers by using a solvent in which the polymer (or pre-polymer in the case of insoluble polymers such as polyimide) is soluble. Such silicate layers, counting on weak forces that maintain the layers united can be easily dispersed in an adequate solvent by the entropy increase caused by the disorganization of the layers which exceeds the organizational entropy of the lamellas.

Polymer is, then, adsorbed in the delaminated layers, and when the solvent is evaporated (or the mixture is precipitated), layers are reunited, filled with polymer to form, in the best case, an ordered multilayer structure, that is, an intercalated morphology.

Moreover, there is melt intercalation. In this method, silicate is mixed with the thermoplastic polymer matrix in its melt state. Under these conditions and if the layer surface is

sufficiently compatible with the chosen polymer, polymer chains can be dragged to the interlamellar space and form an intercalated or exfoliated nanocomposite.

The driving force in the melt intercalation process is the enthalpic contribution of the interactions between polymer and clay. The advantage of this technique is the non-use of solvent (Ke & Stroeve, 2005; Souza et al., 2006).

Finally, a less common method is also employed, in which a clay slurry is directly mixed with the polymer. The idea of this technique consists in increasing the basal spacing of the clay in the first stage of the process, by the polymerization of the monomers or clay suspension in water/solvents and hence, the polymer matrix is intercalated in the increased basal spacing (Kaneko et al., 2007).

5. Clay and polymer modifications for nanocomposite formation

In some cases, polymer or clay is not chemically compatible and there is thus the need of chemical modification of these materials for nanocomposite formation. Two commonly used techniques are: the use of a compatibilizer agent and clay organophilization.

Frequently, a compatibilizer agent can be a polymer which has chemical compatible nature with the polymer and the clay, by a treatment, such as the grafitization of a chemical element containing reactive groups or copolymerization with another polymer also containing reactive groups, compatibility is provided between the materials that will form the nanocomposite (Ke & Stroeve, 2005).

Clay organophilization aims at a better interaction of the clay with the polymer, consisting of the exchange of interlamellar cations (generally sodium and calcium ions) by organic molecules with positive charges that neutralize the negative charges of the silicate layers, aiming to produce an organophilic clay. Generally, amino acids (Usuki et al., 1993), alkylammonium and other salts of this family are used and improve clay wettability by the polymer matrix, facilitating the penetration of the organic molecules between their platelets (De Paiva et al., 2008; Leite et al., 2010).

6. Clay-containing polysulfone nanocomposites

Researches into clay-containing polysulfone nanocomposites began to appear in the literature in 2001. After that, an expressive number of papers and patents have been published in several specialized journals. Next, examples of the scientific advances related to these materials will be presented, according to the following division: polysulfone (PSf), poly(ether sulfone) (PES) and derivatives of polysulfone.

6.1 Polysulfone-clay nanocomposites

In the literature, some papers about PSf-clay nanocomposites are presented. The first one, published in 2001, is about the preparation of a polysulfone nanocomposite with montmorillonite organically modified with dodecylammonium bromide by solution dispersion in dimethylacetamide. Exfoliated nanocomposites were produced and had an improved thermal stability and mechanical properties, although, according to the authors, these two properties were not considerably improved since polysulfone is a high performance polymer with very good properties and it is harder to improve them. Moreover, authors proposed to perform other tests in the future in order to evaluate

different properties, such as, for example, corrosion and diffusion properties (Sur et al., 2001).

Therefore, another study which used solution dispersion, investigated the corrosion properties of a polysulfone nanocomposite membranes prepared with Kunipia-F montmorillonite organomodified with dodecylamine. Low clay contents (as 1 wt.%) produced nanocomposites with superior anticorrosion and barrier properties as a result of the dispersed silicate layers which hinder the diffusion path of oxygen and water (Yeh et al., 2004).

A patent also showed the possibility of preparing polysulfone nanocomposites with smectite clay organomodified with quaternary ammonium ions by two types of organophilization: clay dispersion in water or extrusion of the dry clay with the organomodifier. Nanocomposite preparation was performed by melt intercalation in several types of machines such as extruders, rolls mills, Banbury® type mixers and Banbury® continuous mixers. Features such as improved tensile modulus, tensile strength, gas barrier and heat distortion temperatures could be obtained by creating intercalated or exfoliated mixtures (Ross & Kaizerman, 2003).

A blend composed of PSf, cyanate ester and organophilic MMT was prepared by melt intercalation. Although the flexural modulus was not significantly modified, thermal stability fracture toughness and fracture energy were improved in comparison with the two matrixes due to the good adhesion level of PSf particles to the matrix as well as the homogenous dispersion of the clay mineral platelets into the system (Mondragón et al., 2006).

In 2006, polysulfone nanocomposite membranes were again prepared by solution dispersion in NMP by using an unmodified (Cloisite Na) and organically modified (Cloisite 30B and 93) clays. Only the use of Cloisite 30 B promoted the nanocomposite formation, with an exfoliated/ intercalated morphology. This structure led to enhanced mechanical properties and wettability, while this structure also allowed its use in ultrafiltration operations (Monticelli et al. 2006).

Another nanocomposite, composed of polysulfone and epoxy, with a montmorillonite organomodified with cetyltrimethylammonium bromide was produced by solution dispersion. Homogeneous exfoliated nanocomposites with very improved mechanical properties were obtained (Rajasekaran et al., 2008).

Polysulfone nanocomposites were prepared by contacting a polymeric antimicrobial agent with clay (e.g. Cloisite Na⁺) and a posterior dispersion of the clay platelets in the polysulfone matrix for many applications (Gao et al., 2008).

Sulfonated polysulfone, produced by sulfonation with chlorosulfonic acid, was used with laponite in the preparation of a nanocomposite proton exchange membrane by solution dispersion. It was found that proton conductivity, an important feature in proton exchange membrane, was increased by 25% without affecting water retention, allowing its use as a fuel cell (Lixon-Buquet et al., 2008; Lixon-Buquet et al., 2010).

In 2005, the preparation of polysulfone-clay nanocomposite membranes for filtration area was initiated at the University of São Paulo, Brazil. Since membrane technology has an important role in separation techniques, such as water and wastewater treatment, chemical, metallurgical and petrochemical-related industries, food industries and bioseparation area, special attention should be given to the preparation of membranes, especially the polymer membranes which are largely used in these separation processes. After researches with the

preparation of polysulfone membranes, performed in the beginning of 2000, it was found that the hydrophilicity of the membranes was low and could cause a reduction in water flux and the formation of fouling onto membrane surface. Therefore, an alternative should be found to improve this property.

Techniques such as the use of plasma and sulfonation reactions are used to improve this feature. However, besides enhancing hydrophilicity, mechanical and thermal properties are harmed by using these approaches. Therefore, it was necessary to investigate a technique that could improve membrane wettability without harming other material properties.

Once nanocomposite technology is known to be effective in the improvement of several properties, it was chosen for the preparation of a more hydrophilic membrane. Since sodium montmorillonite from Wyoming is a hydrophilic clay, this clay was then used in the preparation of polysulfone nanocomposite membrane.

The wet-phase inversion method, firstly developed by Loeb and Sourirajan (Loeb & Sourirajan, 1962), is extensively used in the preparation of polymer membranes and consists of the dissolution of the polymer in an appropriate solvent, followed by casting onto a glass plate and immersion of this apparatus-containing the polymer film in a non-solvent bath, where an exchange between solvent and non-solvent will take place, producing membrane coagulation and formation. A combination of solution dispersion, from nanocomposite technology, with the last step of the wet-phase inversion method, that is, the immersion in a non-solvent bath, was used to produce the polysulfone-montmorillonite membranes.

The chosen polymer was polysulfone Udel® P-1700, kindly supplied by Solvay Advanced Polymers; the solvent was N-methyl-2-pyrrolidone and the non-solvent was distilled water at room temperature. Sodium montmorillonite from Wyoming (Sigma commercial name), with cation exchange capacity equal to 95 meq/ 100g, was used without any organophilization.

Different clay contents were studied in this research, which were 0.0, 0.5 and 3.0 wt.% and dispersions consisting of these montmorillonite contents and 25 wt.% PSf were prepared under vigorous mechanical stirring. These dispersions were cast onto a glass support and were immersed in distilled water bath. After 1 minute, membranes were completely detached from the glass surface.

Small-angle X-ray diffraction (SAXRD) [40 kV-30 mA; CuK α radiation ($\lambda = 0.154$ nm); at the rate of 0.6°/ min in the range of 1.5 - 25° (2 θ)], transmission and scanning electron microscopies (TEM and SEM, respectively) were used in order to investigate membrane and nanocomposite morphologies.

Thermal properties were evaluated by thermogravimetry (TGA) (25-900°C, air flow of 50 mL/min; heating rate of 10°C/min and sample mass of 5 mg) and mechanical properties were determined by tensile tests, according to standard ASTM D882-02. Water contact angle measurements were performed to quantify membrane hydrophilicity from free energy of hydration given by Young-Dupré equation (Eq. 1):

$$(1 + \cos\theta) \gamma_w^{\text{TOT}} = - \Delta G_{\text{SW}} \quad (1)$$

Where γ_w^{TOT} is the total surface tension of the water, equal to 73 mJ. m⁻² (Lapointe et al., 2005).

From SAXRD results (Table 1), it was possible to note that, since MMT presented a basal spacing of 14.8 Å and both diffractograms showed a basal spacing of 13 Å, there was the formation of intercalated nanocomposites. In order to prove the intercalation of the

polysulfone chains between the silicate layers, membranes were calcinated until 300°C and SAXRD were again performed. Again, the basal spacings of 13 Å were detected, showing that polymer chains were intercalated in these materials and no water molecules, in this last case, the basal spacing would be reduced to 10 Å, which would mean the collapse of the clay mineral layers due to the absence of polymer chains between them (Grim & Güven, 1978)

Aiming to support SAXRD results, TEM images were obtained. In the membrane prepared with 3.0 wt.% clay, besides PSf intercalation, individual clay mineral platelets could be seen, therefore, showing a complex morphology with partial intercalation/ exfoliation structure (Fig. 3).

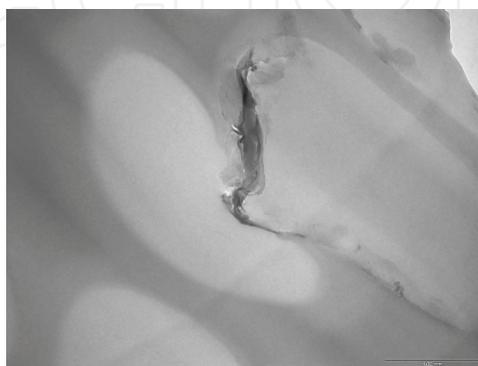


Fig. 3. TEM image of the membrane prepared from the dispersion containing 3 wt.% clay.

The dispersion of the clay particles in the PSf matrix was also evaluated by SEM micrographs (Fig. 4). Clay mineral particles could not be seen in the SEM cross-section micrographs of any of the membranes, which means that there was a good dispersion in the polymer matrix. Moreover, the addition of clay did not promote a change in the cell-like cross-section morphology. Therefore, nanocomposite membranes could be used in the same applications to which PSf membranes were designated.

Clay addition did not change the profile of the TGA curves, either, in comparison with PSf properties (Lisa et al., 2003). Polysulfone nanocomposites presented a two-stage mass loss event around 450°C ascribed to PSf decomposition. From Table 1, it is possible to observe an increase in the onset temperature of decomposition by 3 and 29°C, respectively. This improvement in thermal stability can be explained by the barrier properties conferred by the clay mineral layers which hamper oxygen diffusion into the material and by the labyrinth effect, which is related to the tortuous path the oxygen molecules have to go through. Then, these two combined effects delay volatilization (Lu et al., 2006).

| Clay content (wt.%) | d_{001} of the non-calcinated membranes (Å) | d_{001} of the calcinated membranes (Å) | Onset temperature of decomposition (°C) | Elongation at break (%) | Tensile strength (MPa) | ΔG_{SW} (mJ m ⁻²) |
|---------------------|---|---|---|-------------------------|------------------------|---------------------------------------|
| 0.0 | 0 | 0 | 449 | 31.7±0.7 | 7.8±0.5 | -92.4 |
| 0.5 | 13 | 13 | 452 | 33.8±0.6 | 8.3±0.6 | -95.3 |
| 3.0 | 13 | 13 | 478 | 36.4±0.8 | 9.8±1.1 | -96.1 |

Table 1. Basal spacing (d_{001}) of the non-calcinated and calcinated membranes, onset temperature of decomposition, elongation at break, tensile strength and free energy of hydration (ΔG_{SW}) of the membranes according to clay content.

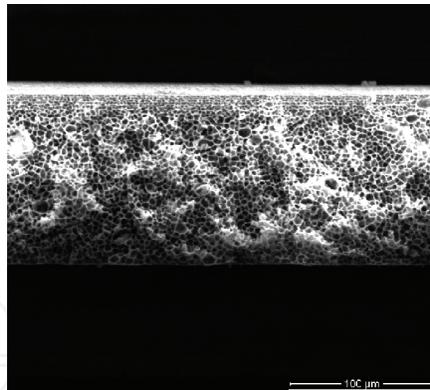


Fig. 4. SEM image of the membrane prepared from the dispersion containing 3 wt.% clay.

An improvement of the mechanical properties could also be observed by the nanocomposite formation, as can be seen in Table 1. Although montmorillonite addition is known to reduce elongation at break (Nigam et al. 2004), this feature was improved in the nanocomposite membranes probably due to a rearrangement of the clay mineral layers in the direction of deformation (Yeh et al., 2004).

Lastly, hydrophilicity was increased with the increase of the clay content and the free energy of hydration values of the nanocomposite membranes were closer to the value of true hydrophilic materials, $\Delta G_{SW} \leq -113 \text{ mJ m}^{-2}$ (van Oss, 1994).

Therefore, a polysulfone nanocomposite membrane, able to be used in filtration operations, with higher hydrophilicity and improved thermal and mechanical properties was obtained by the use of a hydrophilic clay by combining solution dispersion and wet-phase inversion techniques (Anadão et al., 2010).

6.2 Poly(ether sulfone)-clay nanocomposites

Some studies about poly(ether sulfone)-clay nanocomposites can also be found in the literature. The first one was published in 2006 and was related to the investigation of barrier properties of plasma-modified poly(ether sulfone)/ organomodified montmorillonite nanocomposites. Remarkable enhancements in the storage modulus and water/ oxygen permeability were observed in comparison to the pure PES (Wu & Yang, 2006).

The use of attapulgites in the preparation of a nanocomposite with epoxy/ poly(ether sulfone) blends was also studied. The arrangement of attapulgite particles increased the interaction between the epoxy and the poly(ether sulfone) phases and improved the modulus of the material (Zhao et al., 2008).

In 2010, a research was performed concerning a sulfonated poly(ether sulfone) -organophilic montmorillonite nanocomposite proton exchange membrane prepared by solution dispersion with different clay contents. The membrane produced with 3.0 wt.% clay showed the maximum selectivity, $520,000 \text{ S s cm}^{-1}$; besides, all membranes showed a better direct methanol fuel cell performance in comparison with a Nafion® 117 membrane. Other features, such as low cost, convenient processability, reduced methanol permeability, among others, demonstrated the potential of this nanocomposite material to be applied in direct methanol fuel cell applications (Hasani-Sadrabadi et al. 2010).

An epoxy- poly(ether sulfone)-Cloisite 30 B nanocomposite was prepared and characterized by a group from India. The use of two different preparation techniques, ultrasonication and planetary ball milling, led to the formation of distinct morphologies: intercalated and

exfoliated, respectively. Although the addition of clay promoted an improvement in all studied mechanical properties, especially in those with exfoliated morphology, an accentuated decrease in tensile and flexural strength with higher clay contents. Barrier properties were also enhanced and optical transparency was retained until 5 wt.% clay (Asif et al., 2010).

6.3 Derivatives of polysulfone-clay membranes

As far as the author knows, there is only one study about a derivative of polysulfone, poly(arylene ether sulfone), so far. An exfoliated nanocomposite was produced by solution dispersion technique. A higher value of proton conductivity and methanol permeability were obtained in comparison to Nafion® 115, a material widely used in fuel cell membrane. Mechanical properties were also improved in relation to pure polymer. These features showed that these nanocomposites can be used in direct methanol fuel cell operations (Choi et al., 2008).

7. Conclusion

As remarked by the author of the first published paper about polysulfone nanocomposites, as polysulfone plastics are considered high-performance polymers, features such as mechanical and thermal properties are not significantly improved by nanocomposite formation. However, other properties, such as barrier, corrosion and hydrophilicity properties could be improved by the use of the clay nanocomposite technology.

Except for my research, which used sodium montmorillonite, other works showed the extensive use of organoclays in the preparation of these nanocomposites. The most used preparation method was the solution dispersion; therefore, melt intercalation can also be a possibility to produce polysulfone-clay nanocomposite membranes and it could be further investigated in the future. As polysulfone-clay nanocomposites were mainly produced by this technique, which can be used together with the wet-phase inversion method, widely used in membrane preparation, as shown in this author's results, the applications of the nanocomposites presented in the literature is extremely focused on the membrane area, especially in filtration and fuel cell areas. The studies regarding the use of blends composed of polysulfone and other polymer showed that these nanocomposites could be used in the aerospace area.

Finally, having a knowledge developed in these areas, specific improved features can be obtained in the future by using clay nanocomposite technology with different types of clays. Moreover, as mentioned before, the investigation into the use of different techniques to prepare polysulfone can be interesting in order to produce nanocomposites with different properties.

8. Acknowledgment

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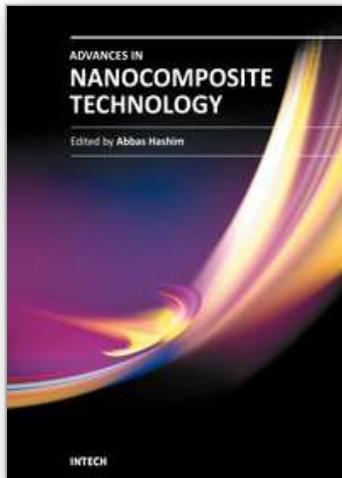
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