

Review

# A Brief Review of the Latest Advances of Attapulgite as a Reinforcing Agent in Polymer Matrix Nanocomposites

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**Abstract:** The development of polymeric nanocomposites using clay minerals as a Nano filler is of great interest to researchers and industry. Many clay minerals are used to modify the properties of the polymers; this strategy improves the thermal and mechanical performance and changes the surface finishing and the processing characteristics. The Attapulgite (ATP), which is a clay mineral of the hydrated magnesium silicates family, has gained prominence in recent years because it combines low cost and high performance. It has a large surface area, strong absorption capacity superior to any other natural mineral, good mechanical resistance and thermal stability. These properties make ATP an ideal candidate for reinforcing polymeric materials. Different approaches and emerging technologies have been applied to improve the thermal and mechanical properties of polymer/ATP nanocomposites which can extend the different chemical treatments used in ATP. Therefore, this review article presents the latest advances related to the use of ATP in the development of polymeric nanocomposites, showing future perspectives for new trends in ATP applications. In general, ATP modifies the mechanical properties of polymers, either in the natural or modified state. And is a good alternative for the replacement of lamellar clays such as montmorillonites with the advantage of having a lower cost and a wide world market to be explored, that which drive new trends in applications for ATP, such as flame retardant of cotton fabrics, dye adsorption, hydrogel membranes for wound dressing, sustainable packaging and fuel cell applications.

**Keywords:** Attapulgite, Nanocomposite, Clay Mineral, Polymers, Thermal and Mechanical Properties

## Introduction

The addition of clay mineral fillers in polymeric materials is a great industrial strategy and has become frequent in the polymer industry. This strategy improves the thermal, mechanical and thermo-mechanical properties, changing the surface appearance and processing characteristics and mainly reducing the final cost of the products (Liu *et al.*, 2020; Gill *et al.*, 2020; Abbasian *et al.*, 2020; Adak *et al.*, 2018; Rapacz-Kmita *et al.*, 2015).

The development of nanocomposites with good cost-benefit resulted in the study of the feasibility and convenience of using a large number of mineral fillers (Rapacz-Kmita *et al.*, 2015). Polymeric nanocomposites are a composite materials class where a dispersed phase has at least one of its dimensions less than 100 nanometers (nm). Thus, the dispersed phase has a large surface area, which allows a greater transfer of tension between the polymer matrix and the dispersed phase. This great transfer of tension results in a high reinforcement notably for the improvement in the mechanical properties,

processability, permeability and thermal stability of the nanocomposite. Another great advantage in the use of nanofillers is the small amount of clay mineral that is used in the composition, contributing for the production of parts with low density and high performance (Bogue, 2011; Hussain *et al.*, 2006; Rane *et al.*, 2018; Thostenson *et al.*, 2005).

Three main methods can be used to obtain nanocomposites with the addition of clay minerals.

**In Situ Polymerization (ISP):** This method involves the addition of the clay filler in the mixture of monomers and polymerization reagents. The nanofillers are swollen by the liquid monomers, followed by the polymerization process. The particles are then surrounded by the polymeric chain that is forming, ensuring the dispersion of the inorganic content. The process is initiated by radiation or heat, in addition to the diffusion of a suitable initiator for the monomer in question. However, very strict control of the polymerization process is required to achieve a good exfoliation of nanofiller (Rane *et al.*, 2018; Alexandre and Dubois, 2000; Liu *et al.*, 2017; Powell and Beall, 2007; Wang *et al.*, 2014; Xie *et al.*, 2014; Zhang *et al.*, 2016).

**Solution Intercalation (SI):** In this method, the clay and the polymer are added to an organic solvent. The solvent is chosen so that the fillers can be easily swollen by it and the polymer dissolves in it as well. The polymer solution will adsorb the clay and disperse it. After that, the solvent is evaporated and the clay particles are well dispersed in the nanocomposite structure (Rane *et al.*, 2018; Alexandre and Dubois, 2000; Giannelis, 1996; Lee *et al.*, 2018; Xiang *et al.*, 2017; Yin *et al.*, 2010a; 2010b).

**Melt Intercalation (MI):** This method consists of increasing the system temperature to melt the polymer and disperse the clay in it. The high shear levels that the polymer chains will exert on the nanofillers cause the clay mineral particles to break and disperse. This way of dispersion of the filler in the polymer is ideal for the properties to be applied in an anisotropic manner, that is, without a specific direction for the stress application. During this process, the melted polymer chains tend to diffuse in between nanofillers to produce the nanocomposite system. This process can be considered eco-friendly, as it does not involve the use of high amounts of solvents. Also, it is the cheapest process because it manages to produce nanocomposites on a large scale (Rane *et al.*, 2018; Zhang *et al.*, 2016; Fornes *et al.*, 2001; Liu *et al.*, 1999; Qi *et al.*, 2013; Zhao *et al.*, 2012).

Polymeric nanocomposites using clay usually have very attractive mechanical and thermal properties and are superior to conventional composites, as well as reduced permeability values, better chemical resistance to solvents and greater flame retardancy. Polymeric composites reinforced with clays are of great interest due to their applications in the packaging and automotive

industries. There are studies on clay reinforced with rubber and silt as well. It is becoming quite common to mix clay and rubber particles processed in different civil and geotechnical constructions such as light landfill, road substrate, fill with embankment, embankments, asphalt construction, sound barrier, railway construction and reinforcement of foundation. However, the use of these mixtures in full-scale projects requires a better understanding of the mechanical performance of the mixtures (Rouhanifar *et al.*, 2020; Majedi *et al.*, 2020; 2021; Yazdani *et al.*, 2018; Rouhanifar and Afrazi, 2019).

Several clay minerals have been widely studied to improve the mechanical properties of polymeric matrices, such as montmorillonite (Rodrigues Passos Severino *et al.*, 2019; Passer *et al.*, 2013a; 2013b; Barbalho, 2012; Bai *et al.*, 2018), halloysite (Bertolino *et al.*, 2020; Lisuzzo *et al.*, 2020; Alam *et al.*, 2020), sepiolite (Ferrari *et al.*, 2017; Kim *et al.*, 2020; Fernández-Barranco *et al.*, 2020; Wang *et al.*, 2020; Sun *et al.*, 2020; Di Credico *et al.*, 2019) and attapulgite (palygorskite) (da Silva *et al.*, 2020; Tian *et al.*, 2020; Yang *et al.*, 2020; Elbassyoni *et al.*, 2020).

Among the existing clay minerals, some have remarkable characteristics like the Palygorskite (Pal) or Attapulgite (ATP). Pal is the term recommended by International Mineralogical Association (IMA) for nomenclature, but the most used trade name for many producers is ATP. In this study, the ATP denomination will be used. This clay has a fibrous morphology with a 2:1 crystalline structure, which consists of two layers of periodically inverted silica tetrahedral connected by an octahedral layer. Each tetrahedral layer presents 180° inversion for each sequence of four or six SiO<sub>4</sub> tetrahedrons. This structure leads to the formation of channels that extend throughout the longitudinal direction of the fiber (Murray, 2020; Frost *et al.*, 2001; López-Galindo *et al.*, 2007).

Bradley (1940) proposed the first structural pattern for ATP and suggested that the clay mineral has the chemical formula [(Mg, Al)<sub>2</sub>Si<sub>4</sub>O<sub>10</sub>(OH) .4H<sub>2</sub>O]. The ATP contains three forms of water in its structure: (a) Coordinated water, with cations of the octahedral leaf, (b) zeolitic water, presents in the channels in which it interacts with both the coordinated H<sub>2</sub>O molecule and the leaf tetrahedral and (c) hydroxyl water, linked to the clay structure in the center of the leaf octahedron (Xavier *et al.*, 2012). Figure 1 shows the chemical structure of the ATP proposed by Bradley (1940).

Due to these structural aspects, ATP has interesting properties such as high specific surface area, high sorption, low surface charges, bleaching power and thixotropic properties in the presence of electrolytes (Haden and Schwint, 1967; Galán, 1996). Some of its properties are listed in Table 1. In addition to these good properties, ATP has a low-cost with a price of 0.2-0.5

US\$/Kg (as a comparison, the price of montmorillonite is on average 10-15 US\$/Kg). A rough cost estimation was made based on the price of materials from the platform (Alibaba.com-09/05/2020).

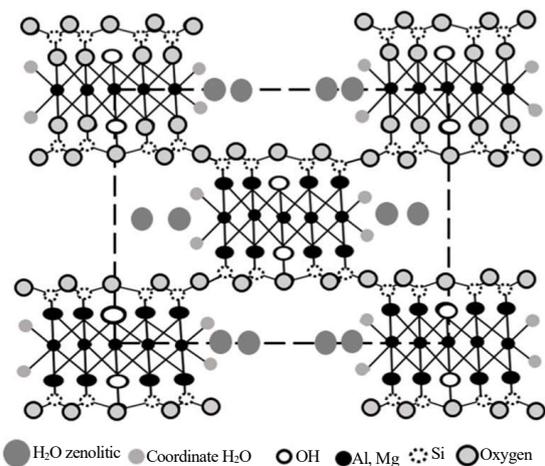
The largest deposits of ATP group minerals around the world were formed either by chemical sedimentation in island seas and lakes, hydrothermal alteration of clays, basaltic glass, or volcanic sediments in the open oceans or by direct crystallization in calcareous soils (Singer and Galan, 2000). These aspects of geological formation are associated with the Mediterranean to semi-arid climate, which reflects in the distribution of these minerals in low latitudes, mainly in arid and semiarid areas of the world (Singer *et al.*, 2011). The location of the main reported deposits of ATP is shown in Fig. 2.

Table 2 shows the chemical composition of ATP from various sources around the world. All the ATPs in the municipality of Guadalupe-PI have clay minerals: Attapulgite, kaolinite, smectite, illite and chlorite, with a predominance of ATP followed by kaolinite. Brazilian ATP has a lower CaO and higher K<sub>2</sub>O content when compared to ATPs from other countries. These differences can be attributed to the content and nature of the Guadalupe ATPs and/or the types and content of contaminants (Baltar and Luz, 2003). The ATP geological formation process allows the association with a large number of accessory minerals, such as quartz, mica, calcite, dolomite, among others, associated with the occurrence of clay minerals; in addition to the presence of organic matter. Often depending on the desired application, it is necessary to separate the main clay mineral from all these impurities (accessory minerals

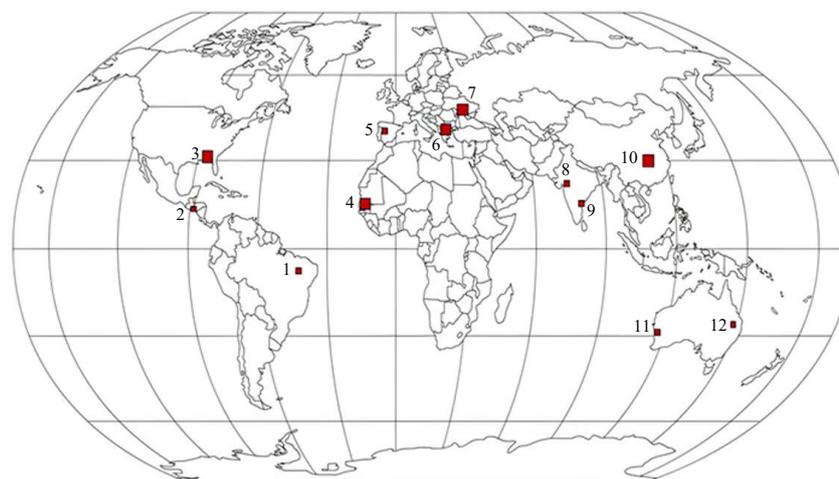
and organic matter) in a process called acid activation (Luz and Lins, 2008).

**Table 1:** Properties of attapulgite (Singer *et al.*, 2011)

Melting point (°C)	1.550
Length (µm)	0.2-2.0
Width (Å)	100-300
Thickness (Å)	50-100
Channels dimensions (Å)	3.7×6.4
Specific surface area (m <sup>2</sup> /g)	150
Cation Exchange capacity (meqiv/100g)	<25
Brookfield viscosity, suspension at 6% in water at 5 rpm (cP)	10.000-12.000
Specific gravity (g/cm <sup>3</sup> )	2.0-2.3



**Fig. 1:** The chemical structure of ATP



**Attapulgite deposits** ■  
 1. Guadalupe, Piauí (Brazil); 2. Guatemala; 3. Meigs-Attapulgius-Quincy District; 4. Theis and Nianming (Senegal); 5. Torrejon and Bercimuel (Spain); 6. Ventzia, Grevena (Greece); 7. Cherkassy (Ukraine); 8. Bhawnagar, Gujarat (India); 9. Maripalli, Andhra Pradesh (India); 10. Guanshan, Anhui (China); 11. Lake Nerramyne (Australia); 12. Ipswich (Queensland)

**Fig. 2:** Attapulgite reported deposits

**Table 2:** Chemical composition of several samples of ATP from different locations (Baltar and Luz, 2003; Grim, 1969; Van Olphen and Fripiat, 1979)

Content (%)	São Pedro Clear Guadalupe Brazil	São Pedro Dark Guadalupe Brazil	Boa Vista Guadalupe Brazil	Nizhnii-Novgorod Russia	Taodeni Saara Algeria	Attapulugus Georgia USA
SiO <sub>2</sub>	68.50	66.70	57.90	51.17	54.71	55.03
Al <sub>2</sub> O <sub>3</sub>	10.30	9.10	12.10	13.73	13.48	10.24
Fe <sub>2</sub> O <sub>3</sub>	4.00	3.70	7.20	1.55	2.10	3.53
FeO	-	-	-	0.31	-	-
MgO	5.60	7.40	4.90	6.40	5.44	0.49
CaO	0.17	0.17	0.10	2.89	2.79	-
K <sub>2</sub> O	1.20	0.83	2.20	-	-	0.47
Na <sub>2</sub> O	0.10	0.11	0.14	-	-	-
H <sub>2</sub> O	-	-	-	10.29	8.65	9.73
MnO	0.12	1.10	0.50	-	-	-
P <sub>2</sub> O <sub>5</sub>	0.03	0.03	0.05	-	-	-
TiO <sub>2</sub>	0.70	0.60	0.61	-	-	-
L.F.*	9.50	11.06	13.37	13.24	12.63	10.13

\* L.F.- Loss to fire

## Purification of ATP

The fibrous morphology, in addition to the presence of the channels, results in a relatively large surface area for this clay mineral. When channels are filled with impurities, the surface area is between 70 to 150 m<sup>2</sup>/g. Once activated, the area ranges from 120 to 210 m<sup>2</sup>/g (Frost *et al.*, 2001; Xavier *et al.*, 2012; Augsburg *et al.*, 1998).

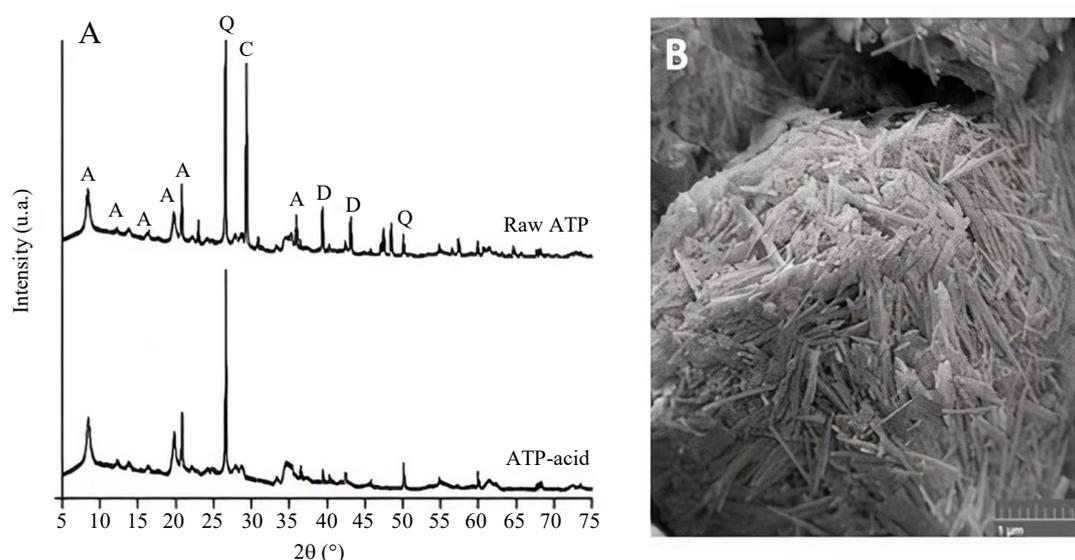
For the removal of quartz and other minerals with larger particle sizes, mainly two methods of physical separation can be used. The first one is the separation by sieving and the second method is the sedimentation technique (Purcell and Parker, 2012). In the first method, the ATP is passed through a sieving process using a 200 mesh (0.074 mm) sieve. For the second method, sedimentation, water-based suspensions with the addition of dispersing agents are required. The most widely used dispersants in the ceramic industry are sodium-based, such as sodium polyacrylate and sodium silicate. Macromolecule sodium polyacrylate has the characteristic of replacing the polar water molecules that are poorly adsorbed in the phyllosilicate sheets, promoting the fluidity of the material. Therefore, the chains generate a double electrical layer, providing high repulsion energy (de Souza *et al.*, 2021). Sodium silicate has the function of neutralizing the reactivity between particles, due to the high surface energies they present in ceramic powders when in a liquid medium. Van der Waals forces act to destabilize the suspensions, forming clusters, in this way, sodium silicate acts in the opposite way towards these forces (Stempkowska *et al.*, 2017). The sedimentation method is based on the difference between the density and/or particle size of minerals to promote separation between them. In this way, a mixing process combined with the action of dispersing agents aims to break up clusters, leaving ATP suspended particles while quartz and other impurities settle in the background. From this, through physical separation methods, non-ATP particles can be removed (Murray, 2000; Purcell and Parker, 2012).

However, only the sieving and the sedimentation technique are not effective to remove the total quartz content, these techniques only reduce the amount of quartz. Many authors have reported the difficulty of completely removing quartz. Neto *et al.* (1993) when analyzing the ATP from Guadalupe (Brazil), submitted the samples to physical processing to reduce the quartz content. Luz *et al.* (1988) through granulometric analysis and X-Ray Diffraction (XRD) found a reduction in the percentage of quartz the finer the granulometry.

In addition to physical separation methods, to minimize other impurities like carbonates and organic matter, a chemical and/or thermal treatment can be used. These treatments also result in an improvement of some properties, such as absorption, adsorption, specific surface area, cation exchange capacity, among others. Chemical treatment can be done by washing or immersing the clay in acid or oxidizing substances. And heat treatment occurs when the sample is heated to a certain temperature and then remains for a certain period (Souza Santos, 1989).

Chemical treatment, also known as acid activation, is very effective for the removal of organic matter and carbonates (limestone and dolomite). It is carried out using oxidizing reagents, such as Hydrogen peroxide (H<sub>2</sub>O<sub>2</sub>) and strong acids, such as Hydrochloric Acid (HCl) and Sulfuric Acid (H<sub>2</sub>SO<sub>4</sub>). H<sub>2</sub>O<sub>2</sub> oxidizes organic matter and depending on the amount of organic matter and the oxidant content is observed a self-combustion (Verdade, 1954).

Using strong acids, the acid activation in the clay occurs as follows: The soluble salts are partially dissolved, resulting in a reduction of iron and aluminum content; sodium and potassium ions go in solution unless they are present into the silicate structure. The calcium ion present is totally or partially solubilized, while the magnesium ion can remain almost entirely in the clay. It is important to note that acid activation allows the improvement of the physical-chemical properties of ATP without destroying its crystalline structure, such as increasing the adsorption capacity, bleaching power and mainly increasing the surface area (Frost *et al.*, 2001).



**Fig. 3:** (a) Diffractogram of raw ATP and ATP-acid and; (b) FEG-SEM micrographs of ATP-acid with magnification of 5000×

An example of the effectiveness of acid treatment in ATP is shown in the study that prepares nanocomposites of Polyurethane (PU)/epoxy blends with natural ATP and ATP-acid (Xu *et al.*, 2019). The results showed that the activation in ATP did not alter the crystalline structures, it improved the glass transition Temperature ( $T_g$ ) of the composites, however, the addition of activated ATP had a significant reinforcing effect on the nanocomposite.

Figure 3a shows the diffractogram of raw ATP and ATP after Acid Treatment (ATP-acid). Analyzing the diffractogram, it is possible to identify four main phases: Attapulgite (orthorhombic structure), calcite, dolomite and quartz. The main ATP diffraction peaks occur at approximately  $8.6^\circ$  and  $19.9^\circ$ . For quartz, the most significant peak is found at  $26.7^\circ$  and for calcite around  $29.3^\circ$ . It is possible to observe that with the acid treatment there was a reduction in the intensity of the peaks of calcite and dolomite, indicating that these impurities are partially removed from the ATP. In addition, the ATP related peaks do not undergo significant changes, which indicates that the acid treatment does not destroy its crystalline structure (Fig. 3b) (Kim *et al.*, 2020; Elbassyoni *et al.*, 2020).

### Modification of ATP

In order to improve the interaction of ATP with polymeric materials, in addition to acid activation, it is desirable to perform a superficial modification of ATP. As this clay mineral has a great capacity for cation exchange, it is possible to perform organophilization by cation exchange. Thus, ATP can be modified through two different processes, silanization with the addition

of aminosilane and organophilization with the addition of an organic compound.

The silanization reactions occur due to the interaction by covalent bonding of the silylating agent with the silanol groups that are on the surface of the clay minerals, as shown in Fig. 4. Silylating agents are compounds that have the general formula,  $(X)_3Si(R)Y$ , in which X is an alkoxide group (RO-), R is called a spacer group which is usually  $(CH_2)_3$  and Y determines the reactivity and applicability of the compound, it assumes several formulas, the most common being  $NH_2$ , Cl, CN, NCO and SH (Xue *et al.*, 2010).

Another technique for the organophilization of clays is by adding organic compounds. The organophilization of clays is usually carried out using the ion exchange technique, or better, with the replacement of cations present in the clay, usually  $Na^+$ . These cations are easily exchangeable because they are monovalent and they facilitate the exchange for organic cations of quaternary ammonium salts (cationic surfactants) or even other types of salts, in an aqueous solution. The salts used in the modification have one or two groups of long-chain hydrocarbons linked directly to a nitrogen atom where the cationic part of the molecule is located (Kakegawa and Ogawa, 2002).

These modifiers allow reducing the surface energy of the clay, improving its wettability with the polymeric matrix. This contributes to the increase in adhesion between the inorganic phase and the polymeric matrix, in addition to facilitating the penetration of the polymeric chains between the clay agglomerates, enabling dispersion and distribution in the polymeric matrix (Xue *et al.*, 2010).

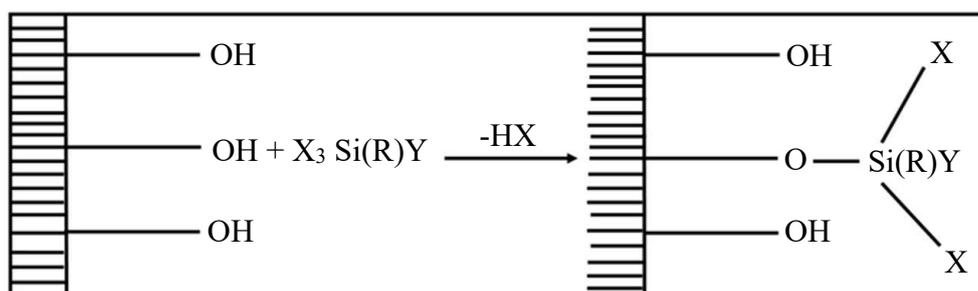


Fig. 4: Scheme of the interaction between the silylating agent and the surface of a clay mineral

## Polymer/ATP Nanocomposites

For an overview of the scientific works related to the use of ATP, an extensive search of the articles and patents was realized on Web of Science and Espacenet databases, respectively. To highlight the most recent works and new trends, the research was limited to the period from 2010 to 2020. Also, to suit the scope of this review, the articles and patents were researched with the specific filters of the use of ATP in nanocomposites of the polymeric matrix. Figure 5a shows the number of articles and patents produced per year and Fig. 5b shows the different processing methods of the polymer/ATP nanocomposites.

Studies on obtaining nanocomposites with the addition of small clay minerals contents to the thermoplastic matrix have shown significant improvement in mechanical properties, water vapor barrier and flammability resistance concerning neat polymer (Gao *et al.*, 2005).

Table 3 summarizes the developments of polymeric nanocomposites-based on a thermoplastic matrix with the addition of ATP, showing the type of ATP treatment, processing method and the highlighted properties.

Thiré *et al.* (2011) developed nanocomposites of PHBV/ATP organically modified with hexadecyltrimethylammonium chloride. The addition of 3 and 5 wt% organophilic ATP led to significant changes in the properties of PHBV like a reduction in the degree of crystallinity and a decrease in the crystalline melting Temperature ( $T_m$ ) and  $T_g$ .

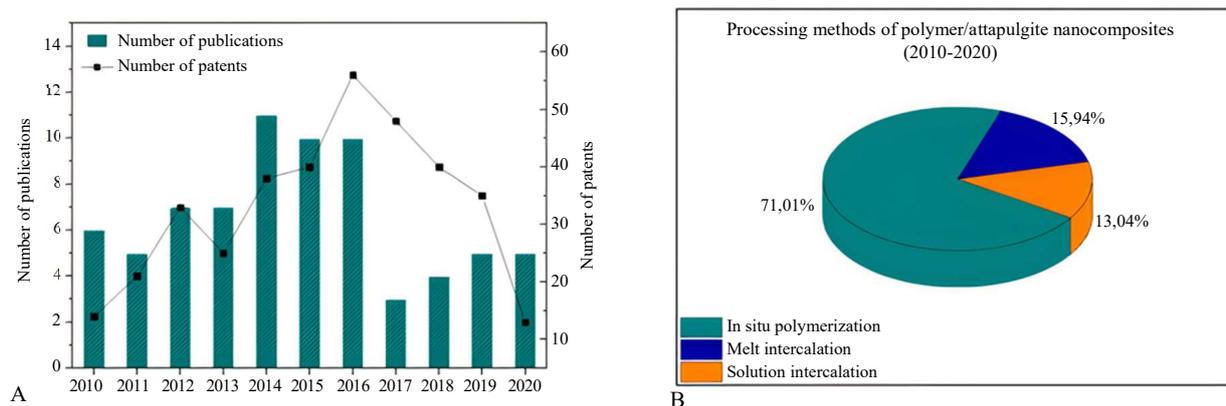
The effect of surface modification of ATP on the morphology and thermal properties of PA6 ATP nanocomposites was studied by Cisneros-Rosado *et al.* (2018). The nanocomposites were prepared by melt intercalation and the ATP was modified with 3-aminopropyl trimethoxysilane bromide and tributylhexadecyl phosphonium. Experimental evidence confirmed the grafting of surface agents into ATP and, as expected, nanocomposites exhibited high thermal stability and less surface energy. The ATP particles favored the formation of the gamma crystalline form and increased the decomposition temperature of PA6.

da Silva *et al.* (2020) prepared PA12/ATP nanocomposites by high-speed mixing using a thermokinetic homogenizer. The addition of ATP increased the modulus of elasticity, hardness, degree of crystallinity and the apparent size of the crystallites. The addition of up to 5% by mass of ATP increased the tensile strength and deformation at rupture, after this amount, the concentration increased significantly and there was no good dispersion.

Wang *et al.* (2014a) prepared PBT/ATP nanocomposites by in situ polymerization. The results show that PBT/ATP nanocomposites have greater thermal stability than neat PBT. The PBT/ATP nanocomposite with a higher ATP content can delay the transport of polymer chains to the growing crystals compared to the nanocomposite PBT/ATP with a lower ATP content. According to dynamic results from the mechanical analysis, the nanocomposite PBT/ATP storage module has been significantly improved and the addition of ATP particles promotes the crystallization of PBT.

In addition to these improvements in mechanical and thermal properties, some recent studies have also studied the influence of ATP on the flammability of nanocomposites. Besides the direct influence of ATP on the flammability of materials, this clay mineral can also be used as a synergetic agent in combination with flame retardants. Hou *et al.* (2020) prepared PA6/Melamine Cyanurate (MCA)/ATP nanocomposites by two-step-melt polymerization. For the composition with 6.2% of ATP and 11.5% of MCA, the mechanical properties of the nanocomposites were up to 44.81 MPa and the samples passed the UL-94 V-0 flammability rating, with the Limited Oxygen Index (LOI) reaching 27.9%. Thus, the authors concluded that the combination of MCA and ATP provided a marked improvement in the flame retardancy of PA6, in addition to maintaining its mechanical properties.

In general, ATP modifies the mechanical properties of polymers, either in the natural or modified state and has been shown to be a good alternative for the replacement of lamellar clays such as montmorillonites with the advantage of having a lower cost and a wide world market to be explored.



**Fig. 5:** (a) number of articles and patents produced per year from 2010 to 2020 and; (b) processing methods of the polymer/ATP nanocomposites

**Table 3:** Summarizes the developments of polymeric nanocomposites-based thermoplastic matrix with the addition of ATP, showing the type of ATP treatment, processing method and highlighted properties.

ATP	Polymer Matrix	ATP content	Processing Method*	Highlighted Properties	Reference
Sieved (320 mesh)	Carboxymethyl cellulose	5, 10, 20 and 25 wt%	ISP	The incorporation of ATP enhanced the swelling capability	Wang and Wang (2010)
Acid activation (HCl)	Polyurethane-imide	Non specified	ISP	This work opens up a possibility for the preparation of infrared low-emissive materials	Wang <i>et al.</i> (2010a)
Sieved (320 mesh)	Hydroxyethyl Cellulose-g-PAA <sup>1</sup>	Non specified	ISP	The introduction of ATP into polymeric network can improve the water absorption rate	Wang <i>et al.</i> (201)
Pretreated with H <sub>2</sub> O <sub>2</sub> and acid activated (HCl)	PAN <sup>2</sup>	1-7 wt%	SI	A small amount of results in a homogeneously stress transfer and energy dissipation, increasing the mechanical properties	Yin <i>et al.</i> (2010a)
Dispersed into ethanol and silane	PP <sup>3</sup>	2-10 wt%	MI	The steady shear viscosities of the nanocomposites are much higher than those of pure PP <sup>3</sup> at lower shear rates	Zhang <i>et al.</i> (2010)
Sieved (200 mesh)	Guar Gum-g-Poly(Sodium Acrylate -co-Styrene)	Fixed	ISP	The composites exhibited improved swelling capacity and rate, pH-resistance, salt-resistance and solvent-responsive properties	Shi <i>et al.</i> (2011)
Pretreated with H <sub>2</sub> O <sub>2</sub> and acid activated (HCl)	PAN <sup>2</sup>	0.5, 1, 3 and 5 wt%	SI	The analysis on the steady shear rheology results showed that elevating temperature may promote the orientation effect of ATP nanorods on the PAN <sup>2</sup> chains	Yin <i>et al.</i> (2010b)
Sieved (200 mesh)	Methylcellulose	5, 10, 20, 30 and 35 wt%	ISP	The nanocomposite shows excellent on-off switching swelling characteristics between pH 2.0 and 7.4	Wang <i>et al.</i> (2010b)
Treated with surfactant	PHBV <sup>4</sup>	1-5 wt%	ISP	The morphology can be adjusted in a wide range by controlling the content of the ATP	Thiré <i>et al.</i> (2011)
Treated with surfactant	PMMA <sup>5</sup>	Non specified	ISP	The different morphologies of ATP/PMMA <sup>5</sup> composite particles can be adjusted in a wide range by simply controlling the introduction of the ATP	Zhang <i>et al.</i> (2012)
Sieved (320 mesh)	Psyllium-g-PAA <sup>1</sup>	5, 10, 20, 30 and 40 wt%	ISP	The nanocomposites possess excellent water absorption in distilled water or saline solutions	An <i>et al.</i> (2012)
Dispersed in ethanol solution	P (MEO <sub>2</sub> MA-co-OEGMA-co-AAc)	Non specified	ISP	The tensile strength, tensile modulus and an effective cross-linked chain density increased with the increasing content of ATP	Wang and Chen (2012)
Purified (not specified) and doped with Fe <sub>3</sub> O <sub>4</sub>	Poly (ethylene glycol)-based hydrogel	1, 3 and 5 wt%	ISP	The hydrogel can continue to swell in an alternating magnetic field after equilibrium swelling in deionized water	Wang <i>et al.</i> (2012)
Sieved (320 mesh)	PBMA <sup>6</sup>	Non specified	ISP	The incorporation of ATP can improve the oil absorbency of PBMA <sup>6</sup>	Wang <i>et al.</i> (2013)
Silane coupling in acid conditions	PP <sup>3</sup>	5, 10 and 15 wt%	MI	The modulus, fracture strength and tensile strength enhanced	Tang <i>et al.</i> (2013)
Grinded	Sodium carboxymethyl cellulose-g-PAA <sup>1</sup>	0-10 wt%	ISP	Excellent pH-responsive behavior and good regeneration ability	Liu <i>et al.</i> (2013)
Natural	PBSA <sup>7</sup>	1, 3, 5 and 7 wt%	MI	Nanocomposites revealed significant improvement in mechanical properties, Especially the break elongation of PBSA <sup>7</sup> /ATP nanocomposite with 1 wt% ATP	Qi <i>et al.</i> (2013)
Natural	PAA <sup>1</sup>	Non specified	MI	The composite showed well-connected open nanoscale channels and pores in its structure	Chen <i>et al.</i> (2013)
Washed with distilled water	PoPD <sup>8</sup>	Non specified	ISP	Pal-PoPD <sup>8</sup> sensor displays excellent analytical performances for glucose determination	Luo <i>et al.</i> (2013)
Washed with distilled water	PmPD <sup>9</sup>	Non specified	ISP	Cr(VI) ions can be efficiently adsorbed onto the PmPD <sup>9</sup> -PG nanocomposite	Xie <i>et al.</i> (2014)
Baked, grinded and acid activated (HCl)	PAA <sup>1</sup>	Non specified	ISP	The microgel had a high absorption capacity towards heavy metal ions, especially to Pb <sup>2+</sup> .	Liu <i>et al.</i> (2014a)
Baked, grinded and acid activated (HCl)	PAA <sup>1</sup>	Non specified	ISP	The ATP/PAA <sup>1</sup> nanocomposite hydrogel showed high uptake toward a cationic dye	Zhu <i>et al.</i> (2014)
Natural	Epoxy	3%	Thermoset Matrix	The simulation result revealed that nanoparticle can indeed change the crack initiation and propagation pattern	Zhang <i>et al.</i> (2014)
Already purified	PBT <sup>10</sup>	1 and 3 wt%	ISP	ATP acts as a heterogeneous nucleating agent in PBT <sup>10</sup> crystallization and accelerated the crystallization rate	Wang <i>et al.</i> (2014a)
Received doped with Fe <sub>3</sub> O <sub>4</sub>	Poly (ethylene glycol) based microgel	1 and 3 wt%	ISP	The microgels possess increasing pH response and excellent temperature-responsive characteristics	Yuan <i>et al.</i> (2014)

**Table 3:** Continue

Natural	PAMPS <sup>11</sup>	Non specified	ISP	The addition of appropriate amount of APT can effectively improve the water absorbency and salt water performance of the superabsorbent composites	Xu <i>et al.</i> (2014)
Baked, grinded and acid activated (HCl)	PAA <sup>1</sup>	15 - 25wt%	ISP	The ATP/PAA <sup>1</sup> hydrogel showed good adsorption selectivity toward the Pb <sup>2+</sup> ion	Liu <i>et al.</i> (2014b)
Silane treatment	PP <sup>3</sup> and LLDPE <sup>12</sup>	3, 5 and 7 phr	MI	Tensile modulus of all compositions increased initially and then decreased, indicating embrittlement of polymer	Panda <i>et al.</i> (2014)
Baked, grinded and acid activated (HCl)	PAA <sup>1</sup>	Non specified	ISP	The nanocomposite hydrogels showed a good selective adsorption to Pb <sup>2+</sup> at pH 5	Jiang and Liu (2014)
Acid activated	<sup>13</sup>	5, 10 15 and 20 wt%	MI	The flame retardancy of the composites was improved	Liu <i>et al.</i> (2014c)
Treated with HCl and H <sub>2</sub> O <sub>2</sub>	PEI <sup>14</sup>	Non specified	ISP	The nanocomposite proved to be a promising candidate for early tumor diagnosis	Han <i>et al.</i> (2014)
Modified with a silane coupling agent	PU <sup>15</sup>	Non specified	ISP	The storage modulus (T <sub>g</sub> ), tensile strength and Young's modulus nanocomposites were enhanced with increased loading	Wang <i>et al.</i> (2014b)
Natural	Chitosan/PVAI <sup>16</sup>	1-6 wt%	SI	Tensile strength increase	Lu <i>et al.</i> (2015)
ATP firstly baked at 140°C; acid activation (HCl)	P(AAc-co-Am) <sup>17</sup>	Non specified	ISP	All nanocomposite hydrogels showed better adsorption selectivity to Cu <sup>2+</sup> and Pb <sup>2+</sup>	Liu <i>et al.</i> (2015)
Natural	PANI <sup>18</sup>	Non specified	ISP	FTIR showed a good polymer coating in the ceramic particles	Chae <i>et al.</i> (2015)
Milled and sieved (320 mesh)	Hydroxyethyl cellulose-based hydrogel	Non specified	ISP	The hydrogel exhibited smart salt and pH responsive behaviors	Shi and Wang (2015)
Milled and sieved (320 mesh)	Epoxy resin	Non specified	Thermoset Matrix	The prepared coating exhibited super hydrophobicity	Yang <i>et al.</i> (2015)
ATP modified with [3-(2-aminoethyl)aminopropyl]trimethoxysilane (Z-6020)	PP-g-MA <sup>19</sup>	0.5-5 wt%	MI	Impact strength and tensile strength increase	Chen <i>et al.</i> (2015)
Natural	PVC <sup>20</sup>	5 phr	MI	The Vicat softening temperature showed an improvement in the thermal stability	Wang <i>et al.</i> (2015)
Washed in HCl and introduction of a silane coupling agent KH-570	PBS <sup>21</sup>	1 - 8 wt%	MI	Tensile strength increased	Zhang <i>et al.</i> (2016)
Centrifugation (1500 rpm) and acidactivation (HCl).	PS <sup>22</sup>	0.5 - 2 wt%	ISP	Higher T <sub>g</sub> and poorer processability	Zhu <i>et al.</i> (2016a)
Acid activation (HCl)	PAA <sup>1</sup>	Non specified	ISP	The material showed an exceptional Ce <sup>3+</sup> adsorption	Li <i>et al.</i> (2016)
Natural	Chitosan	1-4 wt%	ISP	Fracture stress and elastic modulus were both over 5 times more than that of neat polymer	Wang and Chen (2016)
Dispersed in H <sub>2</sub> O/ethanol solution	PEBA <sup>23</sup>	Non specified	SI	CO <sub>2</sub> permeability and CO <sub>2</sub> /N <sub>2</sub> selectivity were improved	Xiang <i>et al.</i> (2016)
Natural	PPy <sup>24</sup>	Non specified	ISP	Storage modulus and antibacterial activity were improved	Zang <i>et al.</i> (2016)
Natural	PAA-co-PVA <sup>25</sup>	Non specified	ISP	High water absorbency in pH range from 4 to 10	Ma <i>et al.</i> (2016)
Acid activation (HCl)	PAA <sup>1</sup>	Non specified	ISP	Excellent regeneration and reusability	Zhu <i>et al.</i> (2016b)
Natural	Chitosan/PVAI <sup>16</sup>	Non specified	ISP	The material can be used in the treatment of wastewater containing low-concentration Cu(II) ions	Wang and Wang (2016)
Dissolved into 250 mL of 0.5 mol/L- $\gamma$ -H <sub>2</sub> SO <sub>4</sub>	PANI <sup>18</sup>	Non specified	ISP	The material can be used as the electrode material in supercapacitors	Xie <i>et al.</i> (2016)
Dispersed into a solution of $\gamma$ -Aminopropyltriethoxysilane in isopropanol	EVA <sup>26</sup>	0.3-7 Phr	MI	Tear strength, peel strength and compression set were improved	Shao <i>et al.</i> (2017)
Natural	PEBA <sup>23</sup>	Non specified	SI	The material can be used for natural gas sweetening or biogas purification	Xiang <i>et al.</i> (2017)
Natural	PLA <sup>27</sup> /PBAT <sup>28</sup>	1-7.5 wt% <sup>a</sup>	MI	Tensile strength and elongation at break increase.	Zhou <i>et al.</i> (2017)
Thermally-Treated	PU <sup>15</sup>	0.5-2 wt%	ISP	Tensile strength and Elastic modulus increase.	Lee <i>et al.</i> (2018)
Attapulgite (TAT)	EVA <sup>26</sup>	Non specified	SI	The pour point temperature was reduced	Tu <i>et al.</i> (2018)
Dispersed in silane coupling agent and organic acid solution.	PASA <sup>29</sup>	Non specified	ISP	The water absorbency and retention and the microstructure were significantly improved	Lu <i>et al.</i> (2018)
Natural	PDMS <sup>30</sup>	0.5-5 wt%	SI	Tensile strength increase	Lee <i>et al.</i> (2018)
ATP nanorods were homogeneously dispersed in Dimethylacetamide	PANI <sup>18</sup>	1.5%	ISP	The composites studied can be used as microwave-absorbing materials	Bai <i>et al.</i> (2020)
Acid activation (HCl)	Regenerated cellulose	5-20 wt%	SI	Tensile strength increased	Wang <i>et al.</i> (2018)
Acid activation (HCl)	PAA <sup>1</sup>	Non specified	ISP	Flame retardant and mechanical properties were enhanced with the introduction of ATP	Gao <i>et al.</i> (2019)
Acid activation (HCl)	Cellulose	Non specified	ISP	Adsorption capacity of the nanocomposite was increased by incorporation of ATP	Chen <i>et al.</i> (2019)
Natural	Cellulose	Non specified	ISP	The anticorrosive property of the waterborne epoxy coating were improved	Nan <i>et al.</i> (2019)
Thermal activation	Epoxy resin	Non specified	Thermoset Matrix	The anticorrosive property of the waterborne epoxy coating were improved	Nan <i>et al.</i> (2019)
Natural	PPy <sup>23</sup> and PS <sup>22</sup>	2-3.3 wt%	ISP	Compression modulus increased	Liu <i>et al.</i> (2019)
Acid activation (HCl)	Chitosan	1-8 wt%	ISP	Tensile strength and elongation at break increase	Hu <i>et al.</i> (2020a)
Acid activation (H <sub>2</sub> SO <sub>4</sub> )	Chitosan	2-10 wt%	SI	strength increase and elongation at break decrease.	Hu <i>et al.</i> (2020b)
Sieved (200 mesh)	PAI <sup>21</sup>	1-10 wt%	MI	Elastic modulus increased with the addition of ATP	Kim <i>et al.</i> (2020)
Acid activation (HCl)	PAA <sup>1</sup>	Non specified	ISP	Increase of flame retardancy when combined with ZnO	Gao <i>et al.</i> (2020)
Acid activation (HCl)	Cellulose	Non specified	ISP	Increase of heavy metal ions absorbance capacity	Chen <i>et al.</i> (2020a)
NH <sub>2</sub> and PMMA grafting	PVDF <sup>32</sup>	1 and 2 wt%	SI	Increase in surface pores and strength and decrease of contact angle	Tian <i>et al.</i> (2020)
Dispersed in H <sub>2</sub> O/ethanol solution	HDPE <sup>33</sup>	2, 4, 6 and 8 wt%	MI	Increase in mechanical properties and flame retardancy	Chen <i>et al.</i> (2020b)
Co-Ni hydroxides grafting	UHMWPE <sup>34</sup>	2 wt%	MI	Decrease in the friction coefficient and wear volume	Meng <i>et al.</i> (2021)
Dispersion in 20% NaCl solution	Cellulose	4 and 12 wt%	ISP	Increase in Cu <sup>2+</sup> ions removal	Ma <i>et al.</i> (2021)

\* ISP: In Situ Polymerization, SI: Solution Intercalation and MI: Melt Intercalation

<sup>1</sup>Poly (acrylic acid), <sup>2</sup>Polyacrylonitrile, <sup>3</sup>Polypropylene, <sup>4</sup>Poly (3-hydroxybutyrate-co-3-hydroxyvalerate), <sup>5</sup>Poly(methyl methacrylate), <sup>6</sup>Poly(butylmethacrylate), <sup>7</sup>Poly(butylene succinate-co-butylene adipate), <sup>8</sup>Poly(o-phenylenediamine), <sup>9</sup>Poly(m-phenylenediamine), <sup>10</sup>Poly(butylene terephthalate), <sup>11</sup>Poly(2-acrylamide-2-methylpropanesulphonic acid), <sup>12</sup>Linear low density polyethylene, <sup>13</sup>Polyamide 6, <sup>14</sup>Polyethyleneimine, <sup>15</sup>Polyurethane, <sup>16</sup>Poly (vinyl alcohol), <sup>17</sup>Poly (acrylic acid-co-acrylamide), <sup>18</sup>Polyaniline, <sup>19</sup>Maleic anhydride grafted polypropylene, <sup>20</sup>Poly (vinyl chloride), <sup>21</sup>Poly (butylene succinate), <sup>22</sup>Polystyrene, <sup>23</sup>Poly (ether-block-amide), <sup>24</sup>Polypyrrole, <sup>25</sup>Poly (acrylic acid)-co-Poly (vinyl alcohol), <sup>26</sup>Ethylene-vinyl acetate, <sup>27</sup>Poly (lactic acid), <sup>28</sup>Poly (butylene adipate-co-terephthalate), <sup>29</sup>Poly (aspartic acid), <sup>30</sup>Polydimethylsiloxane, <sup>31</sup>Polyimide 12, <sup>32</sup>Polyvinylidene fluoride, <sup>33</sup>High-density polyethylene, <sup>34</sup>Ultra high molecular weight polyethylene

## Future Perspectives

The era of nanotechnology has reached several sectors in recent years, encompassing more and more specific materials for different applications. The polymer/ATP nanocomposites listed here highlight the growing search for these materials with excellent properties. In the past three years (2018-2020), some specific applications have been studied, which drive new trends in applications for ATP, such as flame retardant of cotton fabrics (Gao *et al.*, 2019), dye adsorption (Chen *et al.*, 2019; Chen and Zhu, 2019), hydrogel membranes for wound dressing (Sun *et al.*, 2020), sustainable packaging (Wang *et al.*, 2018) and fuel cell applications (Hu *et al.*, 2020a; 2020b).

Chen *et al.* (2019; Chen and Zhu, 2019) prepared a nanocomposite hydrogel based on cellulose and ATP by a facile method. The SEM micrographs showed that nanocomposite hydrogel exhibited a porous structure and rough inner surface and ATP was incorporated inside. The addition of ATP reduced the swelling degree of nanocomposite hydrogel and enhanced its adsorption capacity. Through the results, the authors concluded that the nanocomposite hydrogel prepared could be used in removing dyes from wastewater.

Hu *et al.* (2020a; 2020b) prepared proton exchange membranes composed of chitosan/modified organic ATP for fuel cell applications. The composite membranes exhibited better mechanical property, dimensional and thermal stability compared to the neat chitosan membrane. The proton conductivity of the composite membrane is also increased, the composite membrane with 4% by weight of ATP content exhibited the highest proton conductivity of 26,2 mS cm<sup>-1</sup> at 80°C with 100% relative humidity, which is 25.1% larger than the pure chitosan membrane. These results can explore a simple and green strategy for preparing chitosan-based proton exchange membranes, which have great potential in the application of proton exchange membrane fuel cells.

In addition to this trend of applications observed in the last three years (2018-2020) and limited for the use in nanocomposites, the specific properties of ATP also allow for other promising advanced uses such as support of nanoparticles for sensor devices and high-performing catalysts, new adjuvants for vaccines, clay-biological interfaces for tissue engineering and bioreactor devices (López-Galindo *et al.*, 2007).

## Conclusion

ATP has interesting properties such as high specific surface area, high sorption, low surface charges, bleaching power and thixotropic properties in the presence of electrolytes. These properties make ATP an ideal candidate for reinforcing polymeric materials. Different approaches and emerging technologies have been applied to improve the thermal and mechanical

properties of polymer/ATP nanocomposites which can extend the different chemical treatments used in ATP. ATP mainly modifies the mechanical properties of polymers, increasing the elastic modulus and tensile strength either in the natural or modified state and with low filler contents (even less than 6%). Regarding thermal properties, ATP acts as a nucleating agent, increasing the degree of crystallinity and the T<sub>g</sub> of the polymeric matrices. In this way, ATP is a good alternative for the replacement of lamellar clays such as montmorillonites with the advantage of having a lower cost (0.2-0.5 US\$/Kg) and a wide world market to be explored, which drives new trends in ATP applications, such as flame retardant on cotton fabrics, dye adsorption, hydrogel membranes for dressings, sustainable packaging and fuel cell applications.

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## Author's Contributions

All authors equally contributed in this work.

## Ethics

This article is original and contains unpublished material. The corresponding author confirms that all of the other authors have read and approved the manuscript and no ethical issues involved.

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