Research, Society and Development, v. 14, n. 10, e05141049584, 2025
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A pressure sensitive adhesives emulsion flame retardant free halogen-based

Uma emulsão adesiva sensível à pressão, retardante de chamas e livre de halogênio

Una emulsión adhesiva sensible a la presión, retardante de llama, libre de halógenos

Received: 09/16/2025 | Revised: 09/29/2025 | Accepted: 09/30/2025 | Published: 10/02/2025

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Abstract
In this paper, we aim to investigate factors such as shell adhesion, tackiness, flammability, solids content, shear strength and film formation, which are very necessary in formulation design. The core-shell microcapsules flame retardant is an important additive for increasing fire safety. Moreover, demands of free halogen flame retardants have been increasing to prevent the generation of toxic and corrosive gases during its thermal decomposition. In this research, was produced and evaluated an adhesives emulsion flame retardant free halogen based. Emulsion adhesives were applied onto a fabric flame retardant backing, adhesion increased with the time of emulsion polymerization and showed a slight decrease with increasing acrylic acid (AA). Addition of microcapsule ammonium polyphosphate with glycidyl methacrylate (MCAPP) reduced flame propagation in relation to the pure adhesive. Compounds with 25% of MCAPP showed a reduction in burn rate between 49% and 54%, and, with 10% of MCAPP decreased between 0% and 35% when compared with the pure adhesive. Microencapsulation process of the flame-retardant ammonium polyphosphate is viable and compatible with its interaction with a self-adhesive emulsion polymer and provide flame retardant characteristics to the self-adhesive, in addition to providing benefits such as being free halogen compound, increasing the thermal stability and using a water-based adhesive.
Keywords: Ammonium polyphosphate; Encapsulation; Flame retardant; Pressure sensitive adhesives.

Resumo
Neste artigo, pretendemos investigar fatores como adesão da casca, pegajosidade, inflamabilidade, teor de sólidos, resistência ao cisalhamento e formação de filme, que são muito necessários no design de formulações. O retardante de chamas de microcápsulas núcleo-casca é um aditivo importante para aumentar a segurança contra incêndio. Além disso, a demanda por retardantes de chamas livres de halogênio tem aumentado para evitar a geração de gases tóxicos e corrosivos durante sua decomposição térmica. Nesta pesquisa, foi produzido e avaliado um adesivo em emulsão retardante de chamas livre de halogênio. Os adesivos em emulsão foram aplicados em um suporte de tecido retardante de chamas, a adesão aumentou com o tempo de polimerização da emulsão e mostrou uma ligeira diminuição com o aumento do ácido acrílico (AA). A adição de polifosfato de amônio com metacrilato de glicidila (MCAPP) à microcápsula reduziu a propagação da chama em relação ao adesivo puro. Compostos com 25% de MCAPP mostraram uma redução na taxa de queima entre 49% e 54%, e, com 10% de MCAPP, diminuiu entre 0% e 35% quando comparado ao adesivo puro. O processo de microencapsulação do polifosfato de amônio retardante de chamas é viável e compatível com sua interação com um polímero em emulsão autoadesivo e, confere características retardantes de chamas ao autoadesivo, além de proporcionar benefícios como ser livre de compostos halógenos, aumentar a estabilidade térmica e utilizar um adesivo à base de água.
Palavras-chave: Polifosfato de amônio; Encapsulamento; Retardante de chamas; Adesivos sensíveis à pressão.

Resumen
En este artículo, nuestro objetivo es analizar factores como la adhesión a la superficie, la viscosidad, la inflamabilidad, el contenido de sólidos, la resistencia al corte y la formación de película, que son esenciales para el diseño de la formulación. El retardante de llama de microcápsulas con núcleo-cubierta es un aditivo importante para aumentar la seguridad contra incendios. Además, la demanda de retardantes de llama libres de halógenos ha aumentado para prevenir la generación de gases tóxicos y corrosivos durante su descomposición térmica. En esta investigación, se

produjo y evaluó un retardante de llama de emulsión adhesiva libre de halógenos. Los adhesivos de emulsión se aplicaron sobre un soporte de tela retardante de llama; la adhesión aumentó con el tiempo de polimerización de la emulsión y mostró una ligera disminución con el aumento del ácido acrílico (AA). La adición de polifosfato de amonio en microcápsulas con metacrilato de glicidilo (MCAPP) redujo la propagación de la llama en relación con el adhesivo puro. Los compuestos con 25% de MCAPP mostraron una reducción en la velocidad de combustión de entre el 49% y el 54%, y, con 10% de MCAPP, disminuyó entre el 0% y el 35% en comparación con el adhesivo puro. El proceso de microencapsulación del polifosfato de amonio retardante a la flama es viable y compatible su interacción con un polímero en emulsión autoadhesivo y, proporciona características retardantes a la flama al autoadhesivo, además de brindar beneficios como estar libre de compuestos halógenos, aumentar la estabilidad térmica y utilizar un adhesivo base agua.
Palabras clave: Polifosfato de amonio; Encapsulación; Retardante de llama; Adhesivos sensibles a la presión.

1. Introduction

Self-adhesives formulated with acrylic emulsion polymers or with natural rubber solvent-based adhesives, can have halogenated flame retardants components in your formulations, especially when used in automotive, aeronautics and electronic segments, required to meet legal requirements for safety levels. Most of these flame retardants contain bromine atoms used in their chemical structure, called Brominated Flame Retardants (BFRs), which suffer from thermal degradation and produce halogen radicals (Br°). These halogenated compounds are cheap, effective in the reduction of flammability of products and do not affect adversely the polymer processing capacity (Martins, Valera & Tenorio, 2014). However, they are toxic and are slowly released from the polymer matrix to the environment, making their way to the food chain and eventually, arrive in human contact. Its use inside products was regulated and banned in Europe and some states of the USA, such as through the RoHS Directive (European Commission, 2013) and in also in Europe through the REACH (European Commission, 2014). Therefore, there is a growing demand to prevent the generation of toxic and corrosive gases during thermal decomposition, which has led to the development of free halogen flame retardants (Zhao et al., 2010).

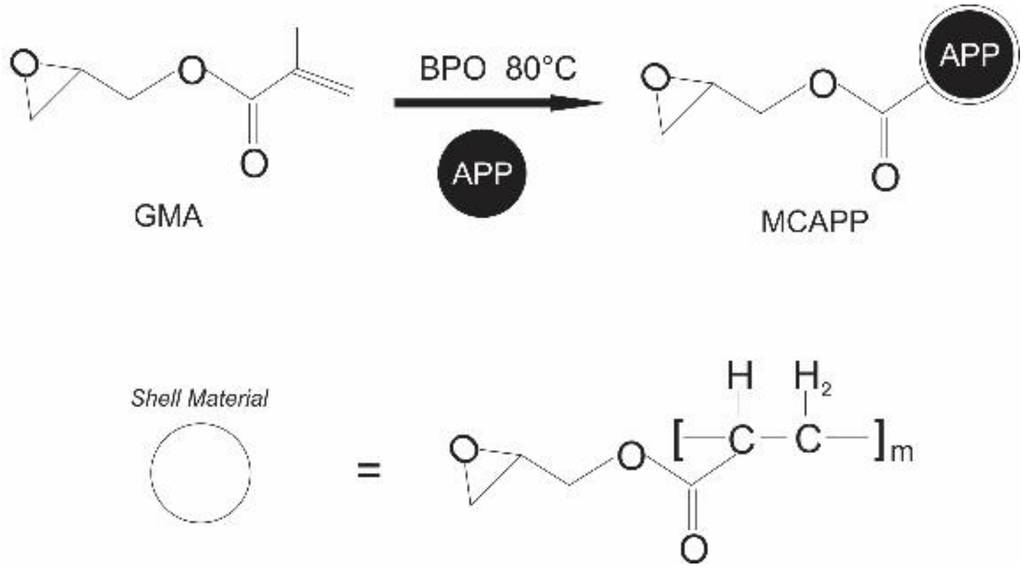
Gower and Shanks (2004) reported in their study that pressure-sensitive adhesives (PSAs) obtained by emulsion polymerization, are composed mainly of butyl acrylate (BA) and/or 2-ethylhexyl acrylate (2-EHA) monomers. However, it is necessary to enhance the glass transition temperature (Tg) of the copolymers formed to increase the shear strength at room temperature, as well as the peel adhesion. To accomplish this enhancement, monomers including methyl methacrylate (MMA), vinyl acetate, styrene and acrylic acid (AA) are added to the polymerization to obtain such effect. The increased monomeric chain of acrylic esters used in the copolymerization with MMA showed an increase in adhesion and copolymerization's with low levels of AA, showed an increase of adhesiveness and tackiness (Gower & Shanks, 2004; Utumi, Corso & Gasparin, 2021). The incorporation of AA into the formulation promotes an increase in the adhesiveness of acrylic polymers, alters surface interactions, modifies rheological behavior and increases tack, reaching its maximum value at AA concentrations between 1.5 and 3.0 phr (Gower & Shanks, 2006; Kowalczyk et al., 2020).

A flame-retardant substance can be defined as one that reduces the flammability of the material in which it is applied. Ammonium polyphosphate (APP) typical inorganic phosphorus flame retardants is a greater non-halogenated flame-retardant substance, however, it has low affinity with polymers, and it is easily affected by water or moisture, causing their migration from the interior to the surface, and reducing their performance capabilities in contact with fire. It is low affinity with polymers causes a loss in the mechanical properties of the composites which are inserted (Liu et al., 2011; Zhao et al., 2014; Breis, Campos & Botton, 2025), then, to overcome these problems the microencapsulation core-shell is a good choice (Tang et al., 2013). When plastic or other materials containing APP are exposed to fire or accidental heat, flame retardant begins to decompose, usually in polymeric phosphoric acid and ammonia (Matsumoto et al., 2021). The phosphoric acid reacts with hydroxyl groups or other synergist groups to form an unstable phosphate ester; the next step is the dehydration of the phosphate ester to form carbon foam (carbonization) on the surface prefixed to the heat source, acting as an insulating layer, preventing further decomposition of the material.

Glycidyl methacrylate (GMA) is a suitable material to use as shell to improve compatibility between the polymer matrix and the flame retardant. GMA monomer has two polymerizable groups, epoxy and methacrylate functions, which can be seen as a modified epoxy resin (Tang et al., 2013).

The schematic diagram of the microcapsule ammonium polyphosphate with glycidyl methacrylate (MCAPP) was presented in Figure 1, obtained with benzoyl peroxide (BPO).

Figure 1. Schematic of microencapsulation reaction of ammonium polyphosphate (APP) with glycidyl methacrylate (GMA) – MCAPP.



Source: Tang et al. (2013).

Therefore, in the present study we used response surface methodology to optimize the production of PSAs. Pressure sensitive adhesives (PSAs) emulsion as a flame retardant free halogen-based was synthesized through the core shell method for use in polymers. The emulsion adhesives were applied onto a fabric flame retardant backing. In this paper, we aim to investigate factors such as shell adhesion, tackiness, flammability, solids content, shear strength and film formation, which are very necessary in formulation design.

2. Methodology

An experimental, laboratory study of a quantitative nature was carried out (Pereira et al., 2018) using simple descriptive statistics with graphs, data classes, mean values and standard deviations (Shitsuka et al., 2014).

2.1 Reagents and suppliers

Ammonium polyphosphate (APP) was supplied by JLS Chemical (USA). Glycidyl methacrylate (GMA) was provided by Vetta Química (Brazil). Cyclohexane was purchased from CAQ – Casa da Química (Brazil). Methyl ethyl ketone (MERK) was provided by Química Moderna (Brazil). Benzoyl peroxide (BPO) was supplied by SP Labor (Brazil). 2-ethylhexyl acrylate, 2-hydroxypropyl acrylate, vinyl acetate, styrene, acrylic acid and sodium persulfate were purchased from Oswaldo Cruz Química (OCQ) (Brazil). All other reagents were of analytical grade and had the purity specifications recommended by the methods used and were obtained from Brazilian suppliers.

2.2 Preparation of microcapsules

The microencapsulation of flame retardant (MCAPP) was prepared using a Tang et al. (2013) procedure. Was used GMA as a shell for the flame-retardant APP. The self-adhesive emulsion was made by emulsion polymerization in a semi-batch process. MCAPP particles were added to the adhesive polymer at the end of polymerization. The adhesive polymers were made according to a 2^3 factorial design, whose factors have been MCAPP concentration, AA concentration relative to the monomer 2-EHA and polymerization reaction time, all of them in two levels.

2.3 Characterization of emulsion

The emulsion characterization process is necessary to distinguish one emulsion from the other. This task is directly related to their performance in the desired application.

2.3.1 Peel Adhesion

Peel adhesion is one of the most important features of a pressure sensitive adhesive, is evaluated by measuring the force required to remove the tape from a surface whose measurement is performed according to literature (Bartlett et al., 2023; Shi et al., 2025).

2.3.2 Tack

Is the adhesive property of forming a bond with a surface after brief contact under virtually no pressure (Shi et al., 2025). The Rolling Ball Tack method was performed according to standard ASTM D3121 (ASTM, 2006).

2.3.3 Shear Strength

This is the ability of a tape to resist static forces applied in the same plane as the surface on which the adhesive was applied (Marin et al., 2023; Breis, Campos & Botton, 2025). The standard PSTC107 (PSTC, 2010) is the most used for this test.

2.3.4 Flammability

To evaluate the burning material was considered that a fire at a location is unlikely to occur when the burn rate of the inner material is zero or very small under the action of a small flame. The flammability tests for adhesive tapes are usually made by ISO 3795 (ISO, 1989).

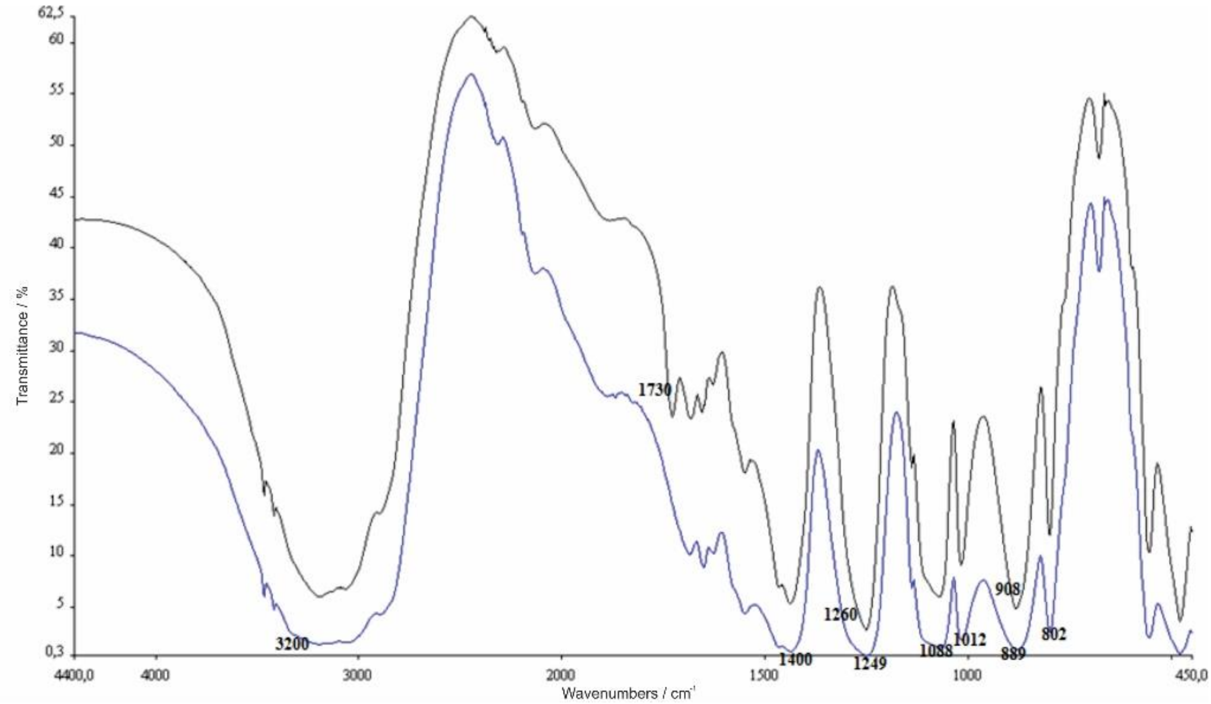
3. Results and Discussion

3.1 FT-IR Spectra

The flame-retardant APP and the microencapsulated MCAPP were analyzed in the infrared region using a FT-IR spectrometer model FrontierTM FT-IR / NIR (Perkin Elmer).

According to Tang et al. (2013), the maximum peak absorption of APP and their corresponding chemical groups include 3200 (N-H), 1249 (P-O), 1088, 1012 and 889 (P-P) cm^{-1}. The MCAPP spectrum shows news absorption peaks at 908, 1260 and 1730 cm^{-1}, corresponding to the grouping stretch C=O acrylate. The double C=C bonds (1640 cm^{-1}) of GMA monomers disappeared, indicating that polymerization occurred, generating a polymer macromolecule adhered to the APP surface. Figure 2 shows the spectra of APP and MCAPP with GMA as shell peaks like those obtained by Tang et al. (2013) and thus it was possible to conclude that microencapsulation was successful.

Figure 2. FT-IR spectra of APP particles and MCAPP particles.



Source: Authors.

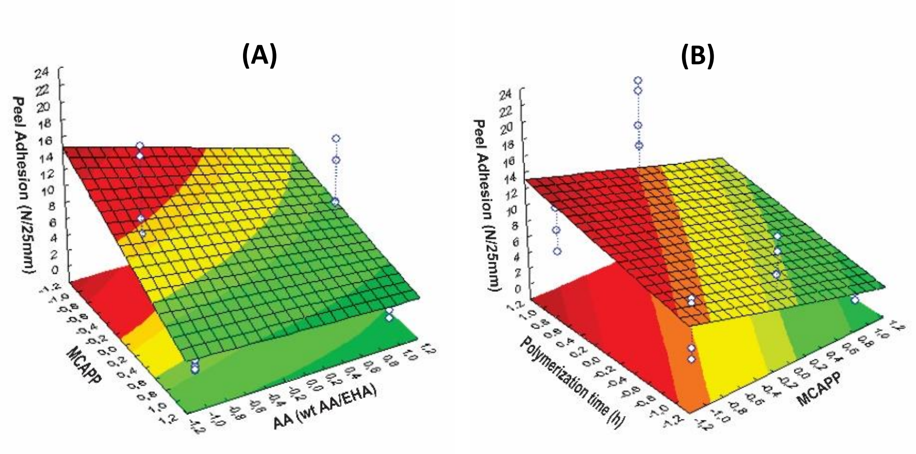
3.2 Peel adhesion

The emulsion adhesives were applied onto a fabric flame retardant backing. The results are shown in Table 1. Peel adhesion was greatly affected by the addition of MCAPP, getting very low values, and the higher the concentration/level of MCAPP the greater the decrease in peel adhesion. According to the results and graphs in Figure 3, the adhesion increased with the time of emulsion polymerization reaction and showed a slight decrease with increasing AA concentration.

According to Gower and Shanks (Gower & Shanks, 2004; Gower & Shanks, 2006), the addition of AA in small portions onto the adhesive increases adhesiveness. However, adhesiveness is impacted negatively when the AA concentration is high, probably because AA increases the viscosity of the final copolymers thus affecting the gel fractions and impacting negatively on the cohesive strength of the adhesive, establishing a connection with the surface onto which it is adhered. Longer polymerization time increased adhesiveness, perhaps because of increased polymer chain, ensuring that the polymerization was completed.

Four MCAPP levels can be noted in Figure 3. It shows that high adhesiveness of the gradient values to be inversely proportional to similar effects. Its negative impact may be due to the fact of MCAPP not being part of the polymer chain, but dispersed in the emulsion, making the cohesive strength of the co-polymers formed over the surface bonded.

Figure 3. Peel adhesion response surfaces. (A) MCAPP x AA concentration; (B) MCAPP x polymerization time.



Source: Authors.

3.3 Tack

The results in Table 1 show that the 25% MCAPP addition reduces the tackiness of the adhesive. It is important to know notice that the tackiness, evaluated by the rolling ball tack and flammability methods, have values inversely proportional to the desired ones, that is, the lower the value the better the expected feature of the analysis.

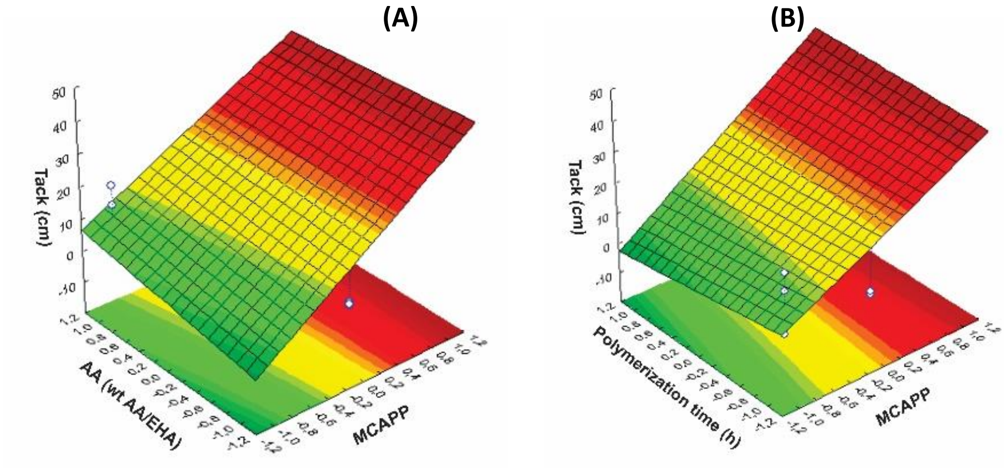
Table 1. Results of tests for PSA pure and PSA/MCAPP composites.

Product	MCAPP (wt%)	AA (wt% AA/EHA)	Polymerization time (h)	Peel adhesion -180º (N/25mm)	Tack (cm)	Shear strength (min)	Flamability (mm/min)	Dripping	Total burn
PSA/MCAPP 1	25	0.0250	2.5	0±0	40±0	0±0	68.125±2.18	No	Yes
PSA/MCAPP 2	10	0.0250	2.5	3.12±1.03	18±4.24	1.5±0.71	149.735±5.48	No	Yes
PSA/MCAPP 3	25	0.0125	2.5	1.125±0.12	40±0	3±0	55.695±1.38	No	Yes
PSA/MCAPP 4	10	0.0125	2.5	9.595±0.40	1±0	15.5±0.71	78.365±3.44	No	Yes
PSA/MCAPP 5	25	0.0250	4.5	1.26±0.38	40±0	2±0	59.555±2.45	No	Yes
PSA/MCAPP 6	10	0.0250	4.5	5.705±1.94	1.25±0.35	26±1.4	115.965±3.73	No	Yes
PSA/MCAPP 7	25	0.0125	4.5	1.91±0.59	40±0	4±0	58.75±5.73	No	Yes
PSA/MCAPP 8	10	0.0125	4.5	10.03±0.38	1±0	30±0	83.315±3.95	No	Yes
PSA 9	0	0.0250	2.5	8.03±0.02	1±0	463±9.9	142.55±3.53	Yes	Yes
PSA 10	0	0.0125	2.5	11.77±1.3	1.75±0.35	401±15.6	119.03±4.48	Yes	Yes
PSA 11	0	0.0250	4.5	14.46±1.82	1±0	91.5±4.95	135.22±3.00	Yes	Yes
PSA 12	0	0.0125	4.5	20.57±0.88	1.25±0.35	79±9.9	128.41±4.68	Yes	Yes

Source: Authors.

From the results presented and the response surfaces in Figure 4, the tackiness was the extreme minimum and maximum to the minimum and maximum levels of MCAPP concentration, respectively, demonstrating an undesired impact on the final product. The concentration of AA and the polymerization time did not show such a high inclination, but AA levels impacted in fall tack (higher value) and the polymerization time increased tackiness (lower value) with the increase in the levels of each factor. As previously reported, according to Gower and Shanks (2004), higher AA levels impact negatively on the tackiness of an adhesive, probably due to the increase in the gel fraction and viscosity of the adhesive.

Figure 4. Tackiness response surfaces. (A) MCAPP x AA concentration; (B) MCAPP x polymerization time.



Source: Authors.

3.4 Shear Strength

As seen from Table 1, all PSA/MCAPP 1 - 8 presented much lower values than those adhesives without flame retardant (PSA 9 – 10). The effect of polymerization times and MCAPP concentration on shear strength were inversely proportional: the lower the polymerization time and concentrations of MCAPP, the greater the time of shear strength.

3.5 Flammability

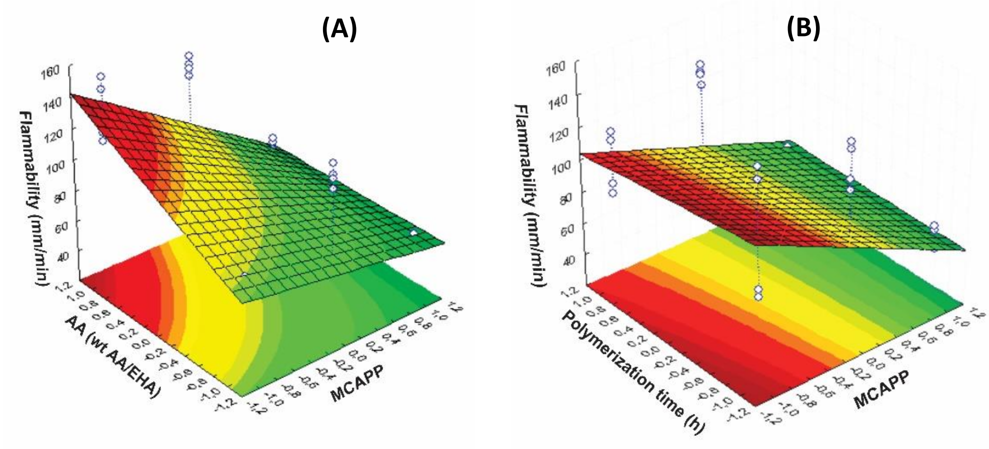
The flammability tests were evaluated using the following parameters: Average speed of flame propagation, the presence of drip and complete burning of the samples (See Table 1).

The products obtained with the addition of MCAPP (MCAPP 1 – 8) did not become flame retardants but had reduced flame propagation in relation to the pure adhesive (MCAPP 9 – 12). The compounds with 25% of MCAPP showed an approximate reduction in burn rate between 49% and 54%, and compounds with 10% of MCAPP showed decreased between 0% and 35% approximately when compared with the pure adhesive. According to Martins et al. (2014), polymeric compounds ABS with 25% of APP showed a reduction in flame propagation speed of 48% compared to pure ABS polymer.

The samples of PSA/MCAPP burned fully but showed no drip or embers after extinguishing the fire. Unlike the adhesive without MCAPP, which burned completely, but had embers and flame drip. Fire drippings are not desirable in flame retardant products (Baraldi et al., 2025). Embers are not mentioned in the methods, but it may be noted that they can influence it negatively.

As shown in Figure 5, the response surfaces for the flammability test were expressed in mm/min (burning rate), the concentration of MCAPP showed high tendency toward flammability based on their maximum and minimum levels. As expected, increasing the concentration of MCAPP reduced burn rate, but not in enough levels to make the tape flame retardant (0 mm/min).

Figure 5. Flammability response surfaces. (A) MCAPP x AA concentration; (B) MCAPP x polymerization time.



Source: Authors.

4. Conclusion

In this study an evaluation of the use of microencapsulated flame retardant on various polymers such as polypropylene (PP), epoxy resins (EP), and styrene-butadiene-acrylonitrile (ABS), among others, but none to use in self-adhesive emulsion polymer, and, on this basis, there is a great opportunity to develop this type of product. The microencapsulation process of the flame-retardant ammonium polyphosphate may become viable and compatible with its interaction with a self-adhesive emulsion polymer and provide flame retardant characteristics to the self-adhesive, in addition to providing benefits such as being free from a compound halogenated, increasing the thermal stability and using a water based adhesive base (solvent free). It also, factors such as peel adhesion, tackiness, flammability, solids content, shear strength and film formation should be considered in the design of the formulation.

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