

Effect of sodium lauroyl sarcosinate on the thermal, optical and moisture absorption properties of polyvinylpyrrolidone film

Efeito do lauril sarcosinato de sódio nas propriedades térmicas, ópticas e de absorção de umidade do filme de polivinilpirrolidona

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Resumo

No presente estudo, filmes de polivinilpirrolidona contendo o surfactante derivado de aminoácido, lauril sarcosinato de sódio (SLS), foram preparados pela técnica de evaporação do solvente. Os filmes obtidos (PVP/SLS) foram caracterizados por espectroscopia de infravermelho por transformada de fourier com refletância total atenuada (ATR-FTIR), propriedades ópticas, análise termogravimétrica (TGA), calorimetria exploratória diferencial (DSC) e por absorção de umidade (AU). Os espectros ATR-FTIR dos filmes de PVP/SLS mostraram as principais bandas do PVP. A opacidade dos filmes de PVP aumentou com a adição de SLS. Além disso, a absorção de umidade dos filmes de PVP/SLS foi maior do que o filme de PVP. As curvas TGA mostraram que a estabilidade térmica do filme de PVP diminuiu após a incorporação do SLS. As curvas de DSC indicaram que SLS pode ser usado como plastificante para PVP.

Palavras-chave: PVP, SLS, plastificante.

Abstract

In the present study, polyvinylpyrrolidone films containing amino acid-based surfactant, namely, sodium lauroyl sarcosinate (SLS), were prepared by casting method. The obtained films (PVP/SLS) were characterized by means of attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR), optical properties, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC) and moisture absorption (MA). ATR-FTIR spectra of PVP/SLS films showed the main bands of PVP. The opacity of PVP films increased with the addition of SLS. Furthermore, moisture absorption of PVP/SLS films were higher than the pristine PVP film. TGA curves displayed that the thermal stability of PVP film decreased after the incorporation of SLS. DSC curves indicated that SLS can be used as plasticizer for PVP.

Keywords: PVP, SLS, plasticizer.

1. Introduction

Several research groups have studied additives for polymers in order to optimize their physical-chemistry properties (mechanical and optical properties, membrane permeability and glass transition temperature- T_g) and to develop eco-friendly materials (Nasrollahi *et al.*, 2022; Tarique *et al.*, 2021; Almeida *et al.*, 2023). The additives for polymers, in general, are organic and inorganic molecules (Marturano *et al.*, 2017) that depending of the production method of polymer can be used in the solid or liquid state (Marturano *et al.*, 2017).

Polyvinylpyrrolidone (PVP) is a water-soluble polymer, biocompatible, non-toxic, inert, thermal resistance, and presents good affinity with drugs (Kurakula and Rao, 2020). Due to these properties, Food and Drug Administration (FDA) considered it as a safe material (Franco and De Marco, 2020). Therefore, PVP is widely used in the areas of pharmacy, cosmetics, food, textile industry, and medicine (Kurakula and Rao, 2020; Franco and De Marco, 2020). Despite its great physical-chemistry properties, PVP is very brittle which makes its thermal processing more difficult (Vaňharová *et al.*, 2022). To remedy this problem, plasticizers such as glycerol, sorbitol, mannitol, arabic gum, polyethylene and glycol have been used during the production of PVP film (Vaňharová *et al.*, 2022). To our knowledge, the use of amino acid surfactants (AASs) as additive for PVP have not yet been investigated.

AASs are a class of surfactants that possess amino acid groups or amino acid residues (Guo *et al.*, 2022) in their chemical structure and can be prepared from agricultural or animal byproducts (Raman *et al.*, 2022). AASs have attracted much attention of the academic community due to the their low toxicity, high biodegradability and antimicrobial activity (Guo *et al.*, 2022). Consequently, AASs are widely used in foods, paints, pharmaceutical products, agrochemicals, and in industrial processes (Guo *et al.*, 2022). Sodium lauroyl sarcosine (SLS) is a type of amino acid-based surfactant ($C_{15}H_{28}NNaO_3$, MW = 293.38 g.mol⁻¹) used in cosmetics, medicated skin cleaners, wound cleaners, soaps, mouthwash, and toothpaste (Bajani *et al.*, 2018; Zhang *et al.*, 2015; Yapar *et al.*, 2017). Furthermore, it is used to promote the methane production, accelerator in the formation of hydrogels and as antimicrobial agent (Zhang *et al.*, 2015; Yapar *et al.*, 2017; Özdemir and Yapar, 2020; Du *et al.*, 2021). The values of critical micelle concentration (CMC) and degree of micelle ionization (α) for SLS at aqueous solution are 12.5ml.dm⁻³ and 0.52 at 298K, respectively (Owoyomi *et al.*, 2014).

The aim this work was to characterize PVP films prepared with different contents of SLS. The effect of addition of SLS on the thermal, optical, and moisture absorption properties of PVP film was investigated by attenuated total reflectance-Fourier transform infrared spectroscopy (ATR-FTIR), UV-vis spectroscopy, thermogravimetric analysis (TGA), differential scanning calorimetry (DSC) and moisture absorption (MA).

2. Experimental

2.1 Materials

The sodium lauroyl sarcosinate (SLS) and PVP (weight average molar mass = 40 Kg.mol⁻¹) were purchased from Sigma-Aldrich. All reagents were used as received. The chemical structures of the molecules investigated are depicted in Figure 1.

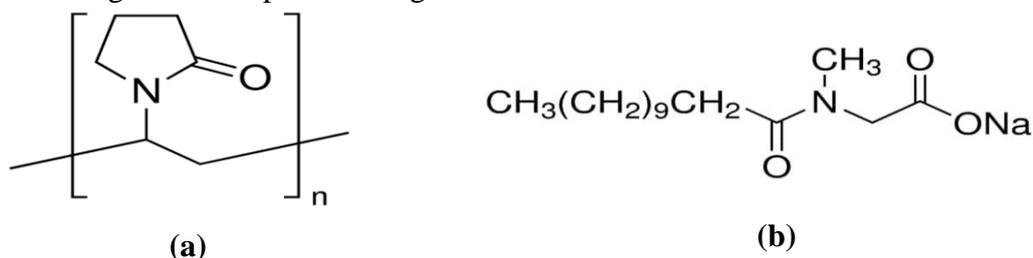


Figure 1 - Schematic representation of chemical structures of (a) PVP and (b) SLS.

2.2 Methods

2.2.1 Sample preparation

Raw PVP and PVP/SLS films were obtained by casting method from aqueous solutions at 40°C. The samples were dried until constant mass. The content of surfactants in the dried mixture with the polymer ranged from 1 to 4 % (w/w). The samples were named as PVP, PVP/SLS 1, PVP/SLS 2, PVP/SLS 3, and PVP/SLS 4 for PVP/SLS films incorporated with 0,1, 2, 3, and 4 % w/w of SLS, respectively.

2.2.2 Attenuated Total Reflectance-Fourier Transform InfraRed Spectroscopy (ATR-FTIR)

ATR-FTIR spectra of pristine PVP and PVP/SLS films were obtained using a Shimadzu IRAffinity-1 spectrometer equipped with a single-reflection attenuated total reflectance (ATR) accessory. A ZnSe crystal mounted in tungsten carbide was used. The analysis was carried out in the frequency range 4000–700 cm⁻¹.

2.2.3 Optical properties of the films.

Transmittance spectra of PVP and PVP/SLS films were recorded in the range of 200 to 800nm using UV-Visible spectrophotometer (Shimadzu, UV2600).

2.2.4 Thermogravimetric Analysis (TGA)

Thermal stability of the samples was performed using a Perkin thermogravimetric analyzer (TGA4000) under dynamic N₂ atmosphere (gas flow of 20 mL/min). The samples were heated in a alumina crucible at a rate of 10°C/min over a temperature range of 30–600°C.

2.2.5 Differential Scanning Calorimetry (DSC)

Differential Scanning Calorimetry (DSC) curves were obtained in Shimadzu equipment. Closed Al crucibles loaded with ~ 3 mg of each sample under a dynamic N_2 atmosphere (50 mL/min) were heated and cooled down at rates of $10^\circ\text{C}/\text{min}$, in the temperature range from 30°C to 200°C . Empty pans were used as reference. The second heating was considered for the determination of the glass transition temperature (T_g) of PVP.

2.2.6 Moisture absorption (MA)

The dried films were stored in closed humidity chamber ($80 \pm 2\%$ relative humidity-RH at $28 \pm 2^\circ\text{C}$) in order to determine moisture absorption. After 24h, the moisture absorption of the films (MA) was calculated using the following equation:

$$MA (\%) = (W_f - W_i) / W_i \times 100$$

where: W_f is the weight of film at time t and W_i is the initial weight of the film.

All films were prepared in duplicate including films without SLS which were used as controls.

3. Results and discussion

3.1 Attenuated Total Reflectance-Fourier Transform InfraRed Spectroscopy (ATR-FTIR)

ATR-FTIR spectroscopy analysis was performed to study the effects of SLS on the structure of the PVP film. Figure 2 shows ATR-FTIR spectra for the pristine PVP and PVP films containing different contents of SLS.

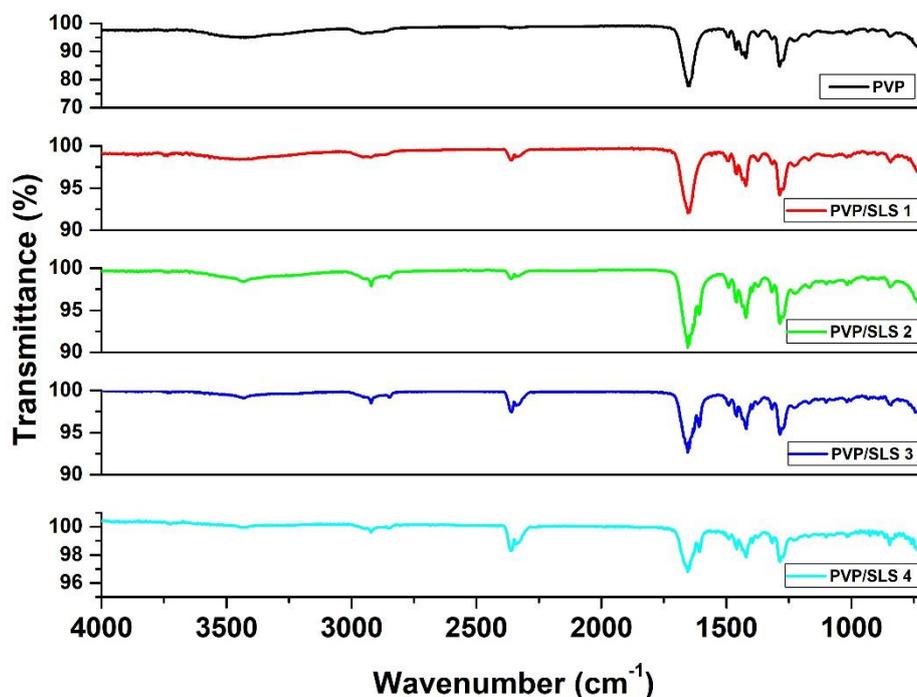


Figure 2- ATR-FTIR spectra of raw PVP and PVP films as a function of SLS content.

The spectrum of PVP film displays main bands at 3442, 2950, 2874, 1644 e 1275 cm^{-1} . These bands can be attributed to the vibrational modes of the groups: OH, CH_2 (asymmetrical stretching), CH_2 (symmetrical stretching), C=O e CN (Safo *et al.*, 2019). The presence of the OH band is due to the hydrophilic nature of PVP (Paula and Mano, 2012). In the case of PVP/SLS films, ATR-FTIR spectra showed all bands of pristine PVP. Besides, it is important to observe in the spectra that after the addition of SLS into polymer film, the intensity of band associated to C=O of PVP decreased, suggesting an interaction between the SLS and the PVP which leads to a decreasing of the amount of carbonyl group in the surface of the film.

3.2 Optical properties of the films

Figure 3 displays the UV-VIS transmittance spectra of pristine PVP and PVP/SLS films in the range of 200 to 800 nm. Pristine PVP film is transparent for visible region. However, after the addition of SLAS, the transparency of PVP film decreased with the rise of SLAS content. Besides, it is important to observe that for PVP/ SLS 1 film, the transmittance was close to the pristine PVP film. Therefore, these results indicate that the incorporation of SLAS content higher than 2% w/w into PVP film alter their morphological structure making it opaque. Furthermore, in the low wavelength region of the UV-VIS transmittance spectra, PVP/SLS films possess higher absorption of the ultraviolet radiation than pristine PVP film, suggesting that SLAS can be used as additive to inhibit, for example, the light-induced deterioration of packaged foods. This property is very important for the food science and to the development of polymer products (Marturano *et al.*, 2017; Dardari *et al.*, 2022).

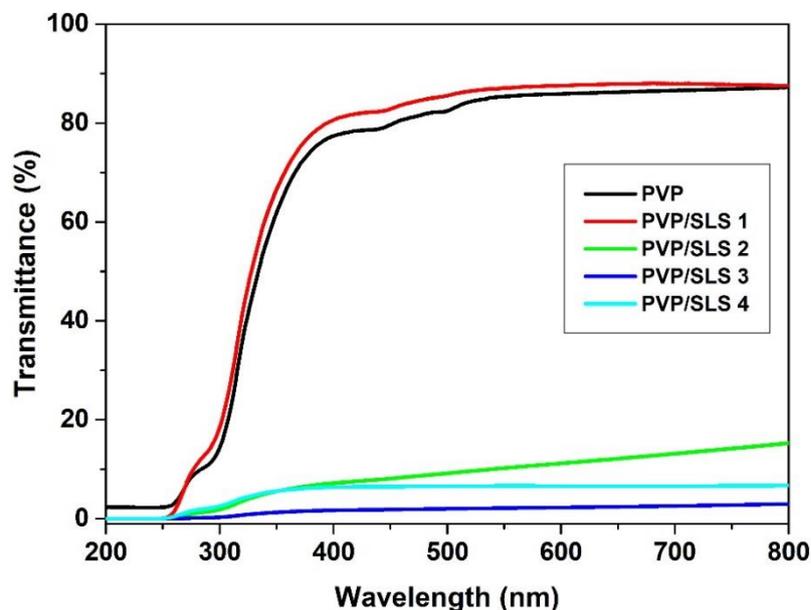


Figure 3 - Transmittance spectra of raw PVP and PVP/SLS films as a function of SLS content.

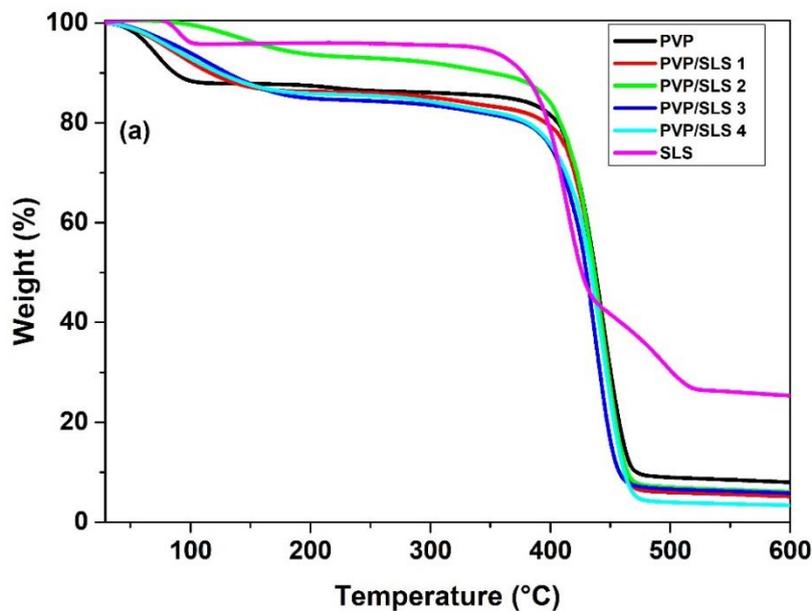
3.3 Thermogravimetric Analysis (TGA)

TGA and DTG curves for pristine PVP and PVP/SLS films are shown in Figures 4 and 5, respectively. The onset thermal decomposition temperature (T_{onset}) and temperature of maximum of mass loss (T_{max}) were determined for each mass loss step of the samples.

For pristine PVP, TGA and DTG curves showed a typical thermal behavior of a polymer. Thermal degradation of PVP occurred in two steps. The first step is associated with the release of water molecules and possesses $T_{\text{max}} = 69^{\circ}\text{C}$. The second step has $T_{\text{onset}} = 406^{\circ}\text{C}$ and $T_{\text{max}} = 444^{\circ}\text{C}$ and is attributed to the thermal degradation of PVP (Haghighat and Mokhtary, 2017). For SLS, it was observed three steps of mass loss. The first step occurred between 50°C and 110°C with $T_{\text{max}} = 90^{\circ}\text{C}$. The second and third steps presented $T_{\text{max}} = 414^{\circ}\text{C}$ and 499°C , respectively. The T_{onset} value for the second step was found at 377°C . Then, T_{onset} values of PVP and SLS show that the thermal stability of PVP is higher than SLS.

For PVP/SLS films, TGA and DTG curves exhibited two steps of mass loss. The first step indicates the liberation of water molecules and the second step show the thermal degradation of PVP and SLS. The T_{max} values for the second step of mass loss of PVP/SLS 1, PVP/SLS 2, PVP/SLS 3 and PVP/SLS 4 films were found at 447 , 442 , 439 , and 445°C , respectively. Furthermore, the T_{onset} values of PVP/SLS films containing 1, 2, 3, and 4 % w/w of SLS were detected at 407 , 400 , 398 , and 400°C .

These results show that TGA curves for PVP/SLS films moves toward lower T_{onset} value with the increase in SLS content. Probably, the presence of SLS into PVP film increase the mobility of the polymer chains (lower interaction between the chains) and hence causes a decrease in the thermal stability of PVP. Voronova et al. reported similar decreasing in the thermal stability of PVP after the incorporation of cellulose nanocrystals (Voronova *et al.*, 2018).



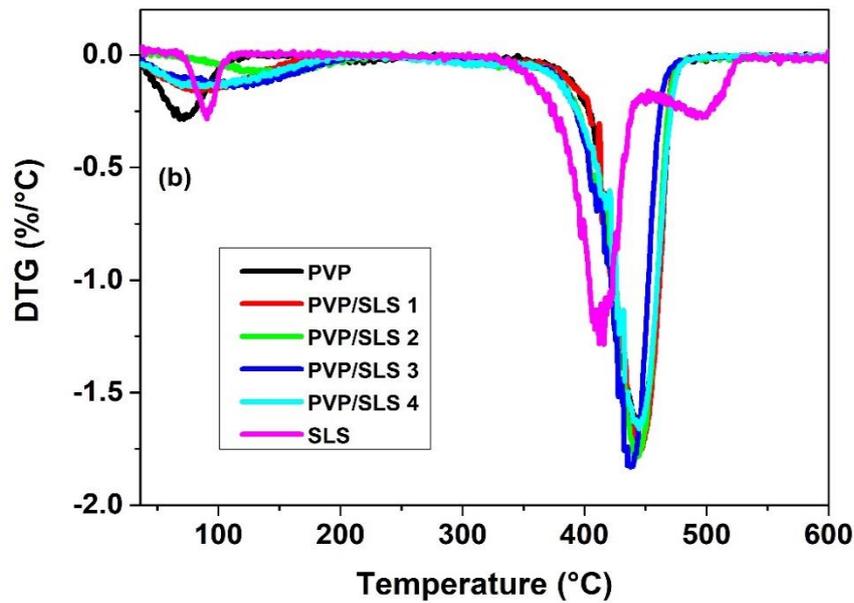


Figure 4- (a) TGA and (b) DTG curves for pristine PVP, SLS and PVP/SLS films.

3.4 Differential Scanning Calorimetry (DSC)

DSC is a technique widely used to investigate thermal properties of polymers such as glass transition temperature (T_g), melting temperature and crystallization temperature (Drzeżdżon *et al.*, 2019). Therefore, DSC curves of pristine PVP, SLS and PVP/SLS films were obtained to study the effect of the incorporation of SL on the thermal behavior of PVP. DSC curves for all samples are illustrated in Figure 5.

PVP exhibited a single glass transition temperature (T_g) at approximately 163°C. This T_g value is in accordance with the literature (Teodorescu and Bercea, 2015). In the case of the SLS, DSC curve showed two endothermic events at around 131°C and 141°C that can be associated to the melting process. Different behavior was described by Hwang *et al.*, (2006). The authors found one endothermic event at 145°C for SLS. This discrepancy in the DSC curve for SLS can be explained by presence of crystals of different sizes in the samples. For PVP/SLS films, DSC curves showed a unique T_g value for all the films in the temperature range studied. Besides, DSC curves did not exhibit the endothermic events associated to SLS. There are two hypotheses for this behavior: (i) a decreasing of the crystallinity of SLS in the polymer matrix or (ii) the dilution of SLS in the polymer film did not allow the observation of this thermal event. Figure 6 presents the T_g values for all the obtained films. The T_g values decreased with the increasing of the SLS amount in the films, indicating that SLS interact with the PVP chains rising their mobility and hence decreasing the T_g . This result corroborates with the obtained TGA results in this work. Therefore, SLS acts as a plasticizer for PVP. These results are in agreement with polymer films containing surfactants (Ghebremeskel *et al.*, 2007; Amim Jr *et al.*, 2012).

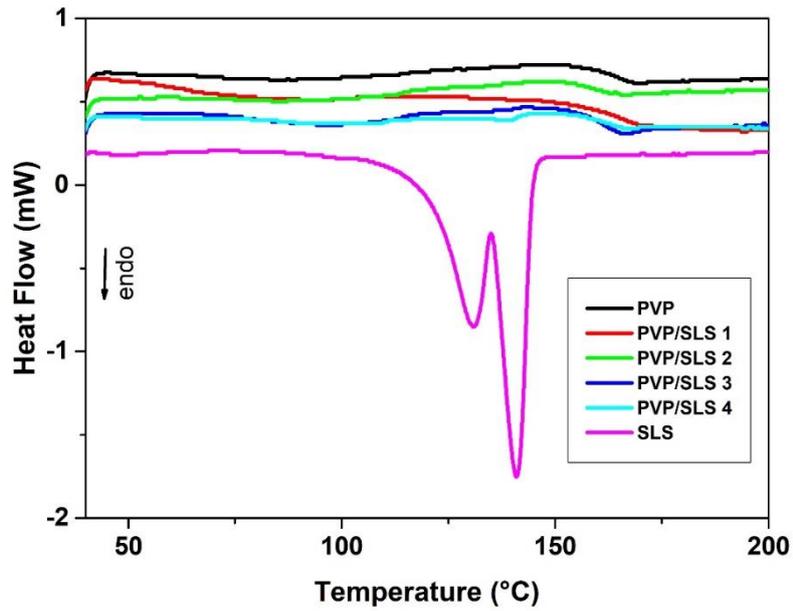


Figure 5- DSC curves for pristine PVP, SLS and PVP/SLS films.

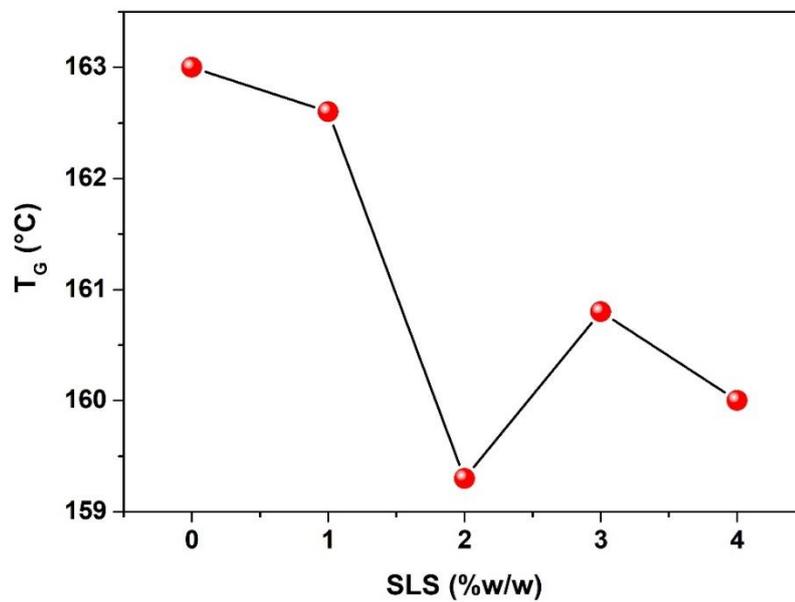


Figure 6 - T_g (°C) values for pristine PVP and PVP/SLS films.

3.5 Moisture absorption (MA)

The MA of the pristine PVP and PVP/SLS films is shown in Figure 7. All the PVP/SLS films have higher MA than pristine PVP film at $(28 \pm 2)^\circ\text{C}$ and $(80 \pm 2)\% \text{RH}$. This result can be explained by presence of higher number of voids or free volume in the PVP film after the incorporation of SLS. As a result, there was an increase of the amount of water molecules diffusing into the PVP/SLS films. Another factor that can have contributed to this, it is the presence of polar functional groups of SLS (such as amino acid group) which favors the formation of new hydrogen bonding interactions with the water molecules. This behavior is consistent with TGA and DSC curves obtained for PVP/SLS films that indicated the plasticizing effect of SLS. Similar result was found for chitosan film containing glycerol as plasticizer (Ma *et al.*, 2019).

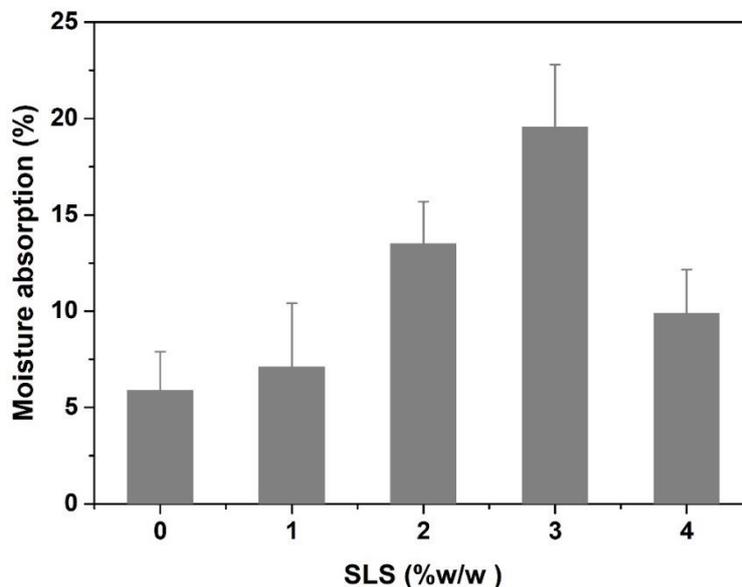


Figure 7- Moisture absorption (%) of PVP films as function of SLS concentration.

4. Conclusions

This work investigated the effect of SLS content on the thermal, optical and moisture absorption properties of PVP film. ATR-FTIR spectra of PVP/SLS films showed a decreasing in the intensity of the carbonyl group of PVP, suggesting an interaction with the SLS which promoted a reorientation of carbonyl group in the surface of the films. Furthermore, the addition of SLS into PVP film increased the opacity and moisture absorption of the obtained films. TGA and DSC curves displayed that SLS acts as plasticizer for PVP film. Therefore, the results indicate that SLS is a promising additive for the development of PVP films.

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