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**Influences of Chemical Alterations on Thermostabilization and Morphology of  
PVC-co-Schiff Base Microspheres**

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**Abstract**

This study focuses on enhancing the thermal stability of Poly(vinyl chloride) (PVC) by introducing Schiff bases, aiming to mitigate thermal degradation. The investigation employs a comprehensive array of analysis techniques, including Fourier Transform Infrared Spectroscopy (FTIR), weight loss estimation, and microscopy (optical, scanning electron, and atomic force) to assess the impact of the modifications. Oven-aging tests reveal notable improvements in color stability for modified PVC films. Results consistently indicate a prolonged color change process in modified PVC, signifying enhanced thermal stability. The modifications demonstrate a remarkable ability to neutralize HCl and passivate labile chlorine atoms; this contributes to elevated thermal resistance and effective prevention of thermal degradation in PVC. This study marks a positive stride towards the development of more stable and reliable PVC materials, with implications for diverse applications demanding superior thermal performance.

**Keywords:** PVC; hydrazide; thermal stability; modification; Schiff base.

**1- Introduction**

Polyvinyl chloride (PVC) has showcased its versatility in various fields such as body protection and healthcare, highlighting its wide range of applications. It's crucial to purify substances both chemically and biologically for certain purposes, which can be effectively achieved through a coordinated approach involving processing, sterilization, and storage in sterile conditions. This adaptable polymer showcases its robustness not just in its inherent physical and chemical traits, but also in its capacity to adjust and react to thoughtfully crafted alterations intended to enhance its interaction with different

substances. These adjustments improve compatibility across various interfaces and minimize the likelihood of adverse reactions that may arise when biologically active fluids come into contact with PVC additives. Achieving this delicate balance involves employing a variety of surface treatments [1].

In recent years, polymer science has seen significant strides in surface modification, resulting in the creation of cutting-edge materials with unprecedented functionalities. Particularly noteworthy are high-performance contrast agents tailored for ultrasound techniques, representing a notable advancement in this domain [2]. Membranes adorned with lipid films affixed to their surfaces have garnered application in biophysics and the production of biosensors [3]. Leveraging both its inherent properties and innovative surface modifications, this polymer is being harnessed to engineer tailored solutions for a diverse array of technical challenges.

In this research, our objective is to chemically alter PVC with the primary aim of extracting heavy metals, a crucial task in environmental preservation to combat metal pollution [4]. Beyond stimulating academic curiosity, the comprehensive endeavors to modify PVC are facilitated by replacing chlorine atoms with a variety of molecular and macromolecular substitutes generated through established organic reactions [5]. These chemical alterations, with roots extending over half a century, have undergone thorough scrutiny, primarily concentrating on dechlorination procedures encompassing both substitution and elimination reactions [6]. Given the unique properties of PVC that make it indispensable across various applications, the necessity of ongoing chemical modification becomes evident. In striving toward this objective, a notable advancement has been witnessed in the utilization of a newly developed polymerization technique,

showcasing its efficacy in surface modification not only of PVC but also of other polymers [7].

The heat treatment of PVC is a widely recognized procedure, used not only for disposal but also for converting plastic waste into molecular compounds, which ultimately leads to the production of a polyene structure. The complex behavior of PVC under elevated temperatures has been the subject of investigation by Yoshioka et al. [8], who employed thermogravimetry coupled with mass spectroscopy (TG-MS) to scrutinize the intricacies of thermal dechlorination/dehydrochlorination. Their comprehensive analysis unveiled the successive formation of a spectrum of molecular compounds, including but not limited to hydrogen chloride (HCl), benzene, propylbenzene, ethylbenzene, ethylmethylbenzene, styrene, xylene, toluene, indane, naphthalene, indene, and methylnaphthalene. The staged pyrolysis process unfolded as follows: (1) HCl production in the first two stages (220-370 °C), indicative of the presence of head-to-head (H-H) and head-to-tail (H-T) units in PVC, (2) exclusive benzene production in the initial stage (220-290 °C) concurrent with HCl, and (3) the development of left aromatics in the concluding stage (> 370 °C).

The novelty of this work lies in the development and application of a single, multifunctional thermal stabilizer for PVC, utilizing Schiff base modification. Traditional methods often require a combination of stabilizers to achieve desired thermal stability and product quality, leading to increased costs and complexity in the stabilization process. This study introduces a more cost-effective and efficient approach to enhancing thermal stability by incorporating Schiff base compounds into the PVC matrix. The innovative aspect is further underscored by the comprehensive investigation of the

modification's effects using a variety of advanced analytical techniques, including SEM, AFM, and thermal analysis.

In alignment with the broader context of PVC modification, this article plunges into the synthesis of Schiff bases employed as thermal stabilizers for PVC at various temperatures. Simultaneously, it embarks on a comprehensive exploration of the ensuing modification effects on PVC, leveraging a diverse array of analytical techniques to unravel the nuanced interplay between chemical modifications and the thermal behavior of this industrially pivotal polymer. In this article, modified PVC by Schiff base is utilized as a thermal stabilizer. The effect of modification was investigated using different techniques.

## **2. Experimental**

### **2.1. Materials**

PVC was obtained from Petkim and served as the foundational polymer in this inquiry, characterized by a K value of 67 and a degree of polymerization of 800. All reagents were employed in their as-received state, with chemicals procured from Sigma Aldrich, UK.

### **2.2. Instrumentation**

FTIR spectra were meticulously acquired utilizing a state-of-the-art instrument (Bruker Alpha ATR-FTIR) spanning the frequency range of  $4000\text{ cm}^{-1}$  to  $400\text{ cm}^{-1}$ . Scanning Electron Microscopy (SEM) and Atomic Force Microscopy (AFM) images were meticulously captured on a TESCAN MIRA3 field emission SEM system

(Kohoutovice, Czech Republic) and a Veeco atomic force microscope (Plainview, NY, USA), respectively. Microscopic examinations were conducted employing the Meiji Techno microscope (Tokyo, Japan). Energy-dispersive X-ray spectroscopy (EDX) measurements were executed leveraging the Bruker XFlash® 6|10 detector (Tokyo, Japan).

### **2.3. Preparation of 4-aminobenzohydrazide:**

A blend containing 3 ml of 80% hydrazine hydrate and 5 ml (equivalent to 0.0399 moles) of ethyl-4-amino benzoate was heated with constant boiling for 4 hr. Afterward, 5 ml of ethanol was added, and the boiling continued for another hour. After cooling, the resulting substance was filtered and further purified through recrystallization utilizing ethanol.

### **2.4. Synthesis of schiff bases**

A blend of the compound with a suitable aromatic aldehyde in 20 mL of absolute ethanol and two drops of glacial acetic acid underwent reflux for 7 hours. The mixture was subjected to cooling to induce precipitation, and the resultant product underwent recrystallization from ethanol.

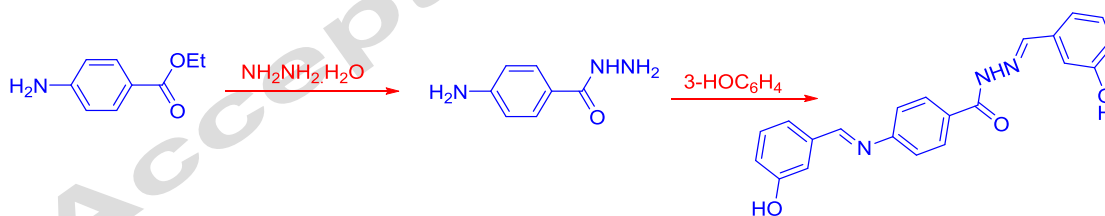
### **2.5. Synthesis of modified PVC polymers**

Modified polyvinyl chloride was synthesized by dissolving 0.1 g of plain PVC in 20 ml of tetrahydrofuran with 0.1 mol of prepared compounds. The reaction mixture was subjected to reflux, and after a duration of 5 hr, the heated mixture was conducted to

casting to facilitate the fabrication of films for the plain and upgraded polymers. The ensuing films were instrumental in elucidating the impact of chemical modifications on the structural and morphological attributes of the PVC matrix.

### 3- Results and Discussion

The synthetic procedure entailed the refluxing of a blend comprising hydrazine hydrate and ethyl-4-amino benzoate. In the subsequent step, 3-hydroxy benzaldehyde, selected as the fitting aromatic aldehyde, was introduced into the reaction mixture dissolved in absolute ethanol. Post-reaction, the mixture underwent a controlled cooling process to instigate precipitation, leading to the generation of the desired yield. The ensuing product was subjected to a meticulous recrystallization process from ethanol, as illustrated in Scheme 1. This sequence of reactions delineates a systematic pathway, ensuring the formation and purification of the targeted compound.

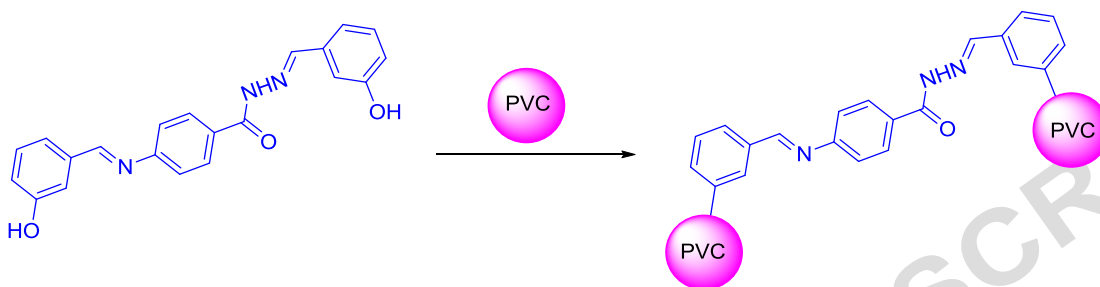


**Scheme 1.** Synthesis of Schiff base.

The reaction between PVC and the Schiff base in tetrahydrofuran (THF) predominantly yields the upgraded PVC, achieving a notably high yield, as depicted in Scheme 2. This transformation signifies the efficacy and selectivity of the reaction process, resulting in the successful incorporation of the Schiff base into the PVC matrix. The utilization of tetrahydrofuran as the solvent underscores its role as a conducive medium for facilitating the reaction and promoting the formation of the desired modified



PVC product. The high yield observed in this reaction affirms the efficiency of the synthetic methodology and suggests its potential applicability for large-scale production of the upgraded PVC material. Table 1 presents the physical properties of synthesized compounds.



**Scheme 2.** Fabrication of the upgraded PVC.

**Table 1.** Physical properties of the prepared compounds.

Compound	Color	Yield (%)	M.P. (°C)
I	White	96%	225-227
II	Orange	81%	217-219
III	Yellow	72%	222-224

### 3.1. FTIR Test for the Prepared Schiff Bases

The FTIR spectra analysis of 4-aminobenzohydrazide, denoted as compound I, discloses discernible changes indicative of the transformation from an ester to an amide structure. Notably, a substantial shift is discerned in the absorption band of the carbonyl group (C=O). Concurrently, the aliphatic C-H peaks vanish. The spectrum manifests prominent peaks at  $3295\text{ cm}^{-1}$  and  $3190\text{ cm}^{-1}$  corresponding to  $\text{NH}_2$  asymmetrical and symmetrical vibration bands, respectively. Signals at  $3150\text{ cm}^{-1}$  for NH,  $3005\text{ cm}^{-1}$  for C-H aromatic, and a recently appeared stretching vibration band at  $1658\text{ cm}^{-1}$  for carbonyl amide were also identified.

For compound II, distinctive bands arise at  $3248\text{ cm}^{-1}$ , signifying the presence of a p-position hydroxyl group. Furthermore, signals at  $3073\text{ cm}^{-1}$  for C-H aromatic, and  $2909\text{ cm}^{-1}$  and  $2880\text{ cm}^{-1}$  for C-H aliphatic are discerned. The spectrum also features a peak at  $1662\text{ cm}^{-1}$  for C=O and an overlap band at  $1628\text{ cm}^{-1}$  for C=N. Importantly, the asymmetric and symmetric  $\text{NH}_2$  signals vanish. Transitioning to compound III, the FTIR spectrum showcases broad bands indicative of the polymer structure. Remarkably, the absorption band of the OH group disappears, while a novel band at  $3243\text{ cm}^{-1}$  emerges, corresponding to NH. However, additional signals include C-H aromatic at  $3073\text{ cm}^{-1}$ , overlap at  $2912\text{ cm}^{-1}$  for C-H aliphatic, C-O-C at  $1320\text{ cm}^{-1}$ , and a band at  $684\text{ cm}^{-1}$  for C-Cl.

Hence, the FTIR spectra yield valuable insights into the structural modifications undergone by the compounds, elucidating alterations in functional groups and affirming the success of the synthesis and modification processes. These spectral analyses provide a nuanced understanding of the chemical transformations, laying the foundation for a comprehensive characterization of the synthesized compounds.

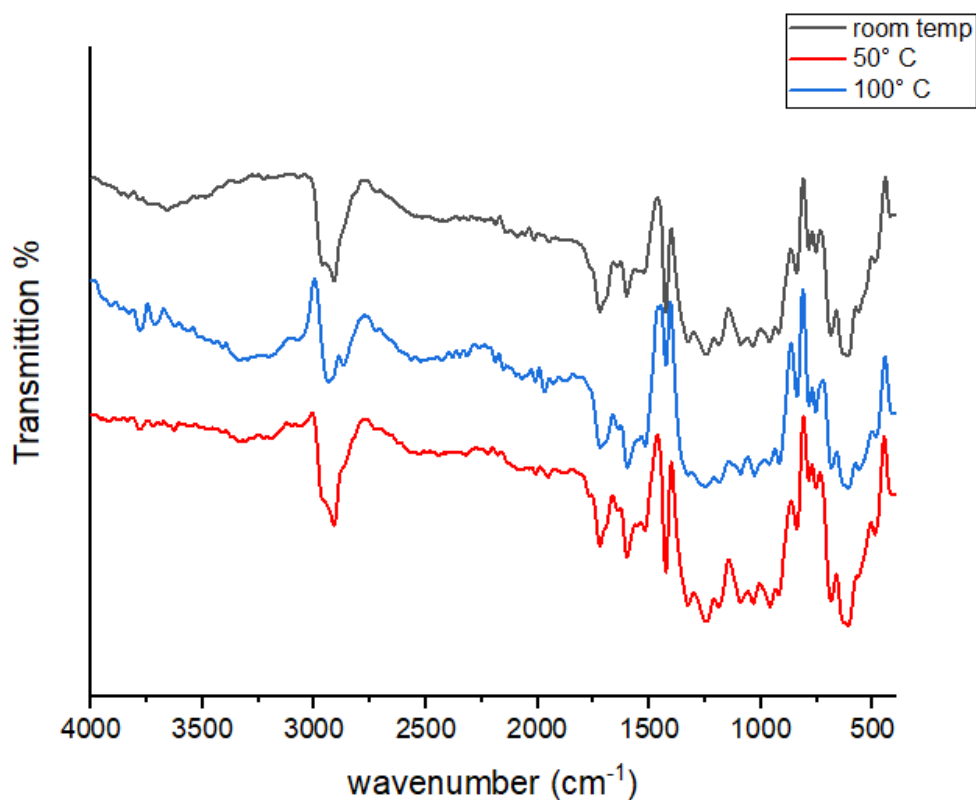
## **3.2. Characterization of the polymeric films**

### **3.2.1. Fourier transform infrared spectroscopy**

The examination of polymeric films, encompassing both unmodified (blank) PVC and modified PVC, entailed a comprehensive analysis of FTIR spectra. The focus was directed towards discerning bending and stretching vibration bands indicative of the chemical composition. These investigations were systematically conducted after subjecting the films to varying temperatures (25, 50, and  $100\text{ }^\circ\text{C}$ ) for 10 minutes.

Remarkably, the intensity of peaks exhibited a discernible temperature-dependent effect, as visually illustrated in Figure 1 and meticulously detailed in Table 2. The variations in peak intensity proffer valuable insights into the temperature-induced alterations in the chemical composition of the films.

This FTIR characterization emerges as an indispensable tool in unraveling the intricate thermal behavior and stability of the polymeric films under diverse temperature conditions. The temperature-dependent changes in peak intensity not only illuminate the dynamic responses of the films to thermal stimuli but also serve as a nuanced fingerprint for discerning and quantifying alterations in the chemical structure of both unmodified and modified PVC films. This comprehensive analysis contributes significantly to the broader understanding of the thermal characteristics and stability profiles of the polymeric films, essential for applications in varying environmental conditions.



**Figure 1.** FT-IR spectra of upgraded PVC sheet after exposure to different temperatures.

**Table 2.** FT-IR spectral data of upgraded polymeric films.

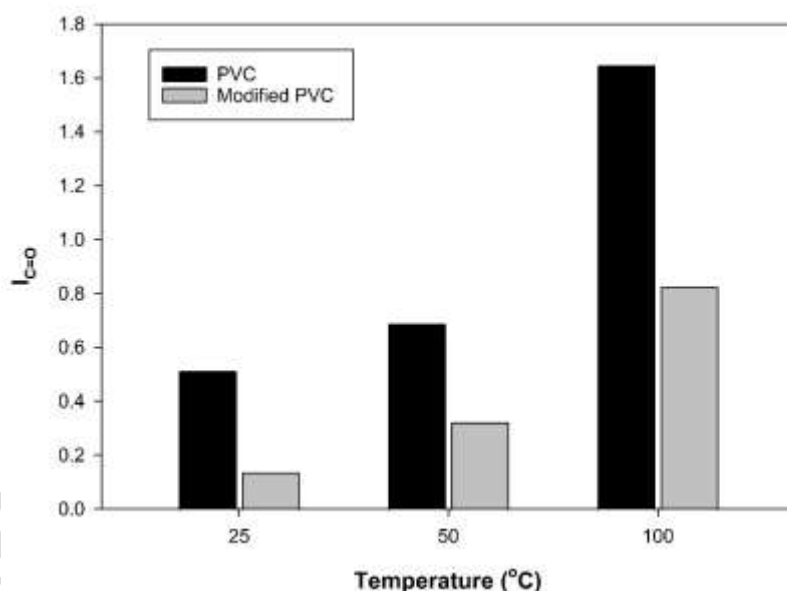
Temp. °C	C=O	C=N	C-H stretch	NH	C-Cl
RT	1723	1644	3071	3348	842
50	1722	1640	3061	3339	843
100	1720	1640	3077	3338	840

FT-IR spectroscopy, spanning the range of 400-4000  $\text{cm}^{-1}$ , served as the cornerstone for scrutinizing alterations in specific functional groups within PVC films. The assessment of thermal stability honed in on monitoring changes in the intensity of the carbonyl peak ( $1722 \text{ cm}^{-1}$ ) concerning a standard peak ( $1328 \text{ cm}^{-1}$ ) under irradiation [9]. The functional group index ( $I_s$ ) was calculated using Equation 1, where  $A_s$  denotes the

absorbance peak during the study, and  $A_r$  represents that of the reference peak [10].

$$I_s = A_s/A_r \dots \quad (1)$$

The carbonyl index ( $I_{C=O}$ ) of PVC sheets was estimated at various temperatures, visually depicted in Figure 2. Each PVC film underwent a singular assessment for elimination and changes in the functional group index. Remarkably, the plain film presented higher pronounced changes in functional index values compared with the upgraded film. These findings significantly affirm the efficiency of Schiff bases as potent thermal stabilizers for PVC.



**Figure 2.** Variation in the  $I_{C=O}$  of modified PVC upon exposure to multiple temperatures.

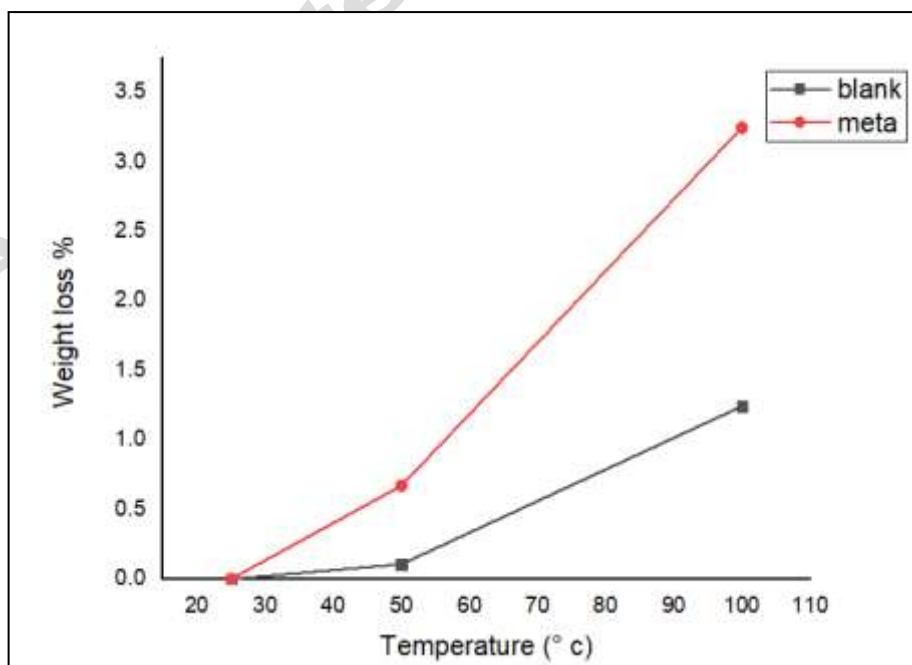
### 3-2-2 Thermal stability and weight loss analysis

Upon exposure to heat, PVC undergoes dehydrochlorination, precipitating the loss of the polymeric material because of the degradation of hydrogen chloride [11]. The strategic utilization of Schiff base serves the dual purpose of modifying and stabilizing PVC against oven-aging degradation. The weight loss percentage was computed using

Equation 2, wherein  $W_0$  denotes the film's weight before heating, and  $W_t$  is the weight after heat exposure.

$$\text{Weight loss (\%)} = [(W_0 - W_t)/W_0] \times 100 \dots \quad (2)$$

The variations in the weight loss percentage of PVC sheets at different temperatures are graphically depicted in Figure 3. Evidently, the weight loss for the blank PVC film surpassed that of the modified one. The modified PVC, in contrast, showcased the least weight loss, underscoring its augmented stability under elevated temperatures. These cumulative findings underscore the pronounced efficacy of the Schiff base in mitigating thermal degradation and preserving the structural integrity of PVC films. The combination of FT-IR spectroscopy and weight loss analysis provides a comprehensive perspective on the dynamic thermal behavior of PVC films, substantiating the utility of the Schiff base as an adept thermal stabilizer in practical applications.



**Figure 3.** Changes in weight loss (%) for modified PVC upon exposure to different temperatures.




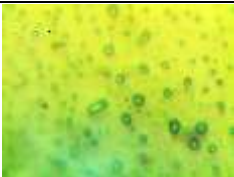


### 3.2.3. Morphology surface of plain and modified PVC films

#### 3.2.3.1. Microscopic test

To evaluate the color durability of PVC samples, we utilized an oven-aging method and studied the ensuing alterations through optical microscopy [12]. The aging procedure revealed differing rates, dependent on the structural makeup of the films. Particularly, noticeable shifts in film colors were detected, linked to the dehydrochlorination process and the emergence of conjugated double bonds within the PVC specimens. The thermal effects on the surface morphology of both PVC and modified PVC films were thoroughly investigated as the temperature increased from approximately room temperature to 100 °C. This assessment utilized an optical microscope with a resolution of 400X. The surface of the modified PVC retained stability when exposed to temperatures ranging from 25 to 100 °C, as detailed in Table 3. This visual observation suggests that the structural modifications introduced by the Schiff base contributed to enhanced thermal stability in the PVC surface, thereby preserving its integrity across a spectrum of temperature conditions.

The optical microscopy analysis not only provides insights into the color stability of the films but also serves as a valuable tool for elucidating the impact of temperature-induced structural changes on surface morphology. The preservation of stability in the modified PVC films underscores the efficacy of the Schiff base in fortifying the PVC matrix against thermal degradation, offering promising prospects for applications demanding color stability and surface integrity under dynamic temperature conditions.

**Table 3.** Microscopic images for polymeric films at different temperatures.

Temp. °C	PVC	Modified PVC
RT		
50		
100		

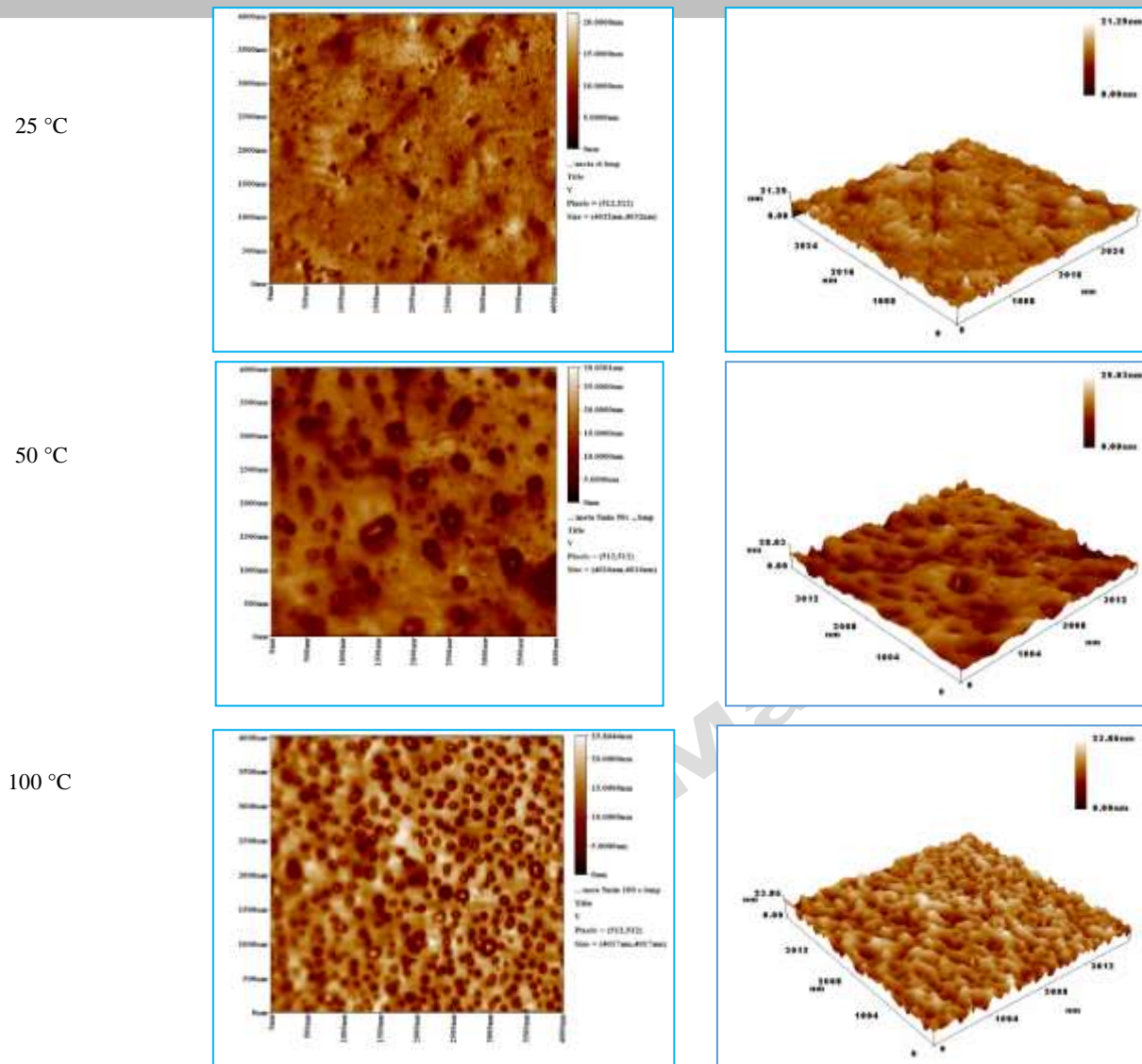
### 3.2.3.2. AFM

AFM is a powerful tool for studying nanoscale material surface morphology and topography. AFM precisely measures surface roughness, texture, and particle size by scanning a sharp tip over a sample. This method is useful for analyzing modified or thermally treated polymers like PVC. AFM's high-resolution images reveal thermal-induced surface changes like roughness and irregularities; it is ideal for studying how dehydrochlorination, chain scission, and crosslinking affect polymer surface thermal stability and integrity. This examination method is essential for characterizing modified PVC films because it accurately assesses thermal stabilizer efficacy. The exploration of thermal effects on surface morphology leveraged the AFM instrument, enabling the examination of both 2D and 3D images of the surface, alongside an assessment of particle size and surface roughness [13]. Prior studies have underscored the efficacy of



AFM in scrutinizing rough surfaces and non-uniform polymers subjected to irradiation.

Table 4 provides a comprehensive overview of the impacts of elevated temperature on particle size and surface roughness properties for blank and modified PVC films. Figure 4 visually portrays an escalation in surface roughness as the thermal temperature is increasing for blank and modified PVC films. These outcomes underscore a notable reduction in the rates of dehydrochlorination and bond-breaking, indicative of the effectiveness of the introduced modifications in fortifying the thermal stability of PVC films. The AFM analysis serves as a pivotal tool, furnishing valuable insights into the morphological alterations induced by thermal exposure. The observed reduction in degradation rates in the modified PVC films aligns coherently with the AFM findings, substantiating the efficacy of the Schiff base in impeding thermal-induced degradation processes and preserving the surface integrity of PVC films. Table 5 compares our findings with other results from the literature.



**Fig. 4.** AFM images for upgraded PVC at 25, 50, and 100 °C.

**Table 4.** Surface features of the upgraded PVC sheet.

Temp. (°C)	Roughness Average (nm)	Particle size (nm)
RT	1.6	73.19
50	3.05	179.83
100	3.04	135.13

**Table 5.** A Comparison of  $R_q$  values with other results in the literature.

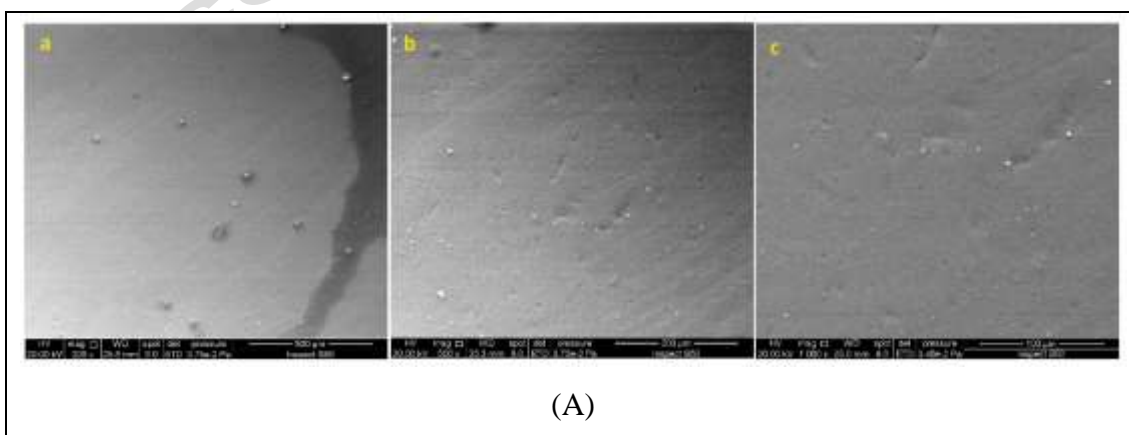
PVC additive	$R_q$	Reference
Schiff bases of 1,2,3,4-triazole-3-thiol	3.3	15
Schiff bases of biphenyl-3,3',4,4'-tetraamine	3.6	16

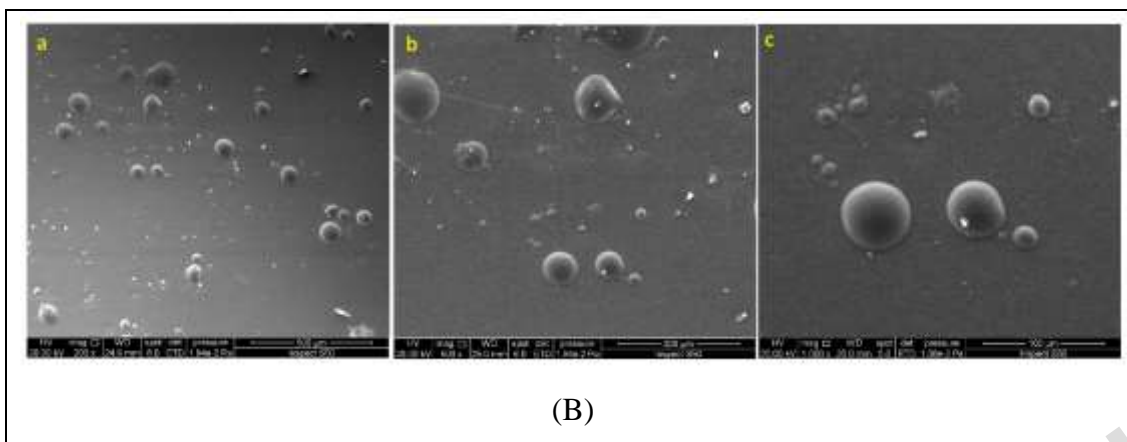
### 3.2.3.3. SEM

The surface morphology of the PVC films, including distortion, variation, particle shape and size, and homogeneity, was examined using scanning electron microscopy (SEM) to obtain detailed information. The morphological changes are mainly caused by dehydrochlorination, chain scission, and crosslinking processes that take place within the polymer matrix. The SEM images provide clear evidence of the roughness and presence of cracks on the unmodified PVC surface following exposure to various temperatures, indicating the occurrence of bond breakage and the elimination of hydrogen chloride. On the other hand, the surface of the modified PVC films exhibited a noticeably smoother and more consistent texture, indicating improved durability and protection against heat-induced deterioration. This analysis highlights the effectiveness of the Schiff base modifications in preserving the structural integrity and surface properties of PVC films when exposed to thermal stress. SEM is harnessed to furnish less distorted, clear, and high-resolution images of particles in materials. This technique proves invaluable in investigating variations in surface properties, cross sections, homogeneity, particle size, and particle shape within blends [12, 13]. The surface morphology of PVC films underwent meticulous scrutiny using SEM. In Figure 5A, SEM images vividly portray the surface of the PVC film after exposure to 50 °C for 10 minutes. The discerned roughness and cracks on the PVC surface, ensuing exposure to different temperatures,

serve as indicative markers of bond breaking inside the polymeric chains and the concomitant elimination of hydrogen chloride. Particularly noteworthy is the comparative analysis of the surface of modified PVC films, which, after exposure to 50 °C, appeared notably smoother and cleaner in contrast to the unmodified PVC.

Figure 5B unveils well-defined spherical shapes of modified PVC at 100 °C under various magnifications. Noteworthy is the persistence of the spherical shape of modified PVC particles even after hypercrosslinking reactions. Antecedent research findings posit that the spherical morphology of modified PVC particles contributes significantly to preventing voids, ultimately yielding a more efficient and reproducible extraction procedure [14]. These SEM analyses contribute valuable insights into the surface characteristics and shape stability of PVC films, thereby robustly supporting the heightened performance of modified PVC under thermal exposure. The observed maintenance of spherical shapes in the modified PVC particles underscores the stability of the introduced modifications, suggesting promising implications for practical applications requiring shape-persistent and thermally stable materials.





**Figure 5.** SEM images for modified PVC with different magnifications at (A) 50 °C and (B) 100 °C.

#### 3.2.3.4. Oven aging for PVC films and color stability

Oven aging served as a method to scrutinize the color stability of PVC films, with observations documented using optical microscopy. The figure succinctly encapsulates the phenomenon of the aging process transpiring at varying rates contingent on the film's structure. As a result, noticeable shifts in film colors were observed, attributed to the dehydrochlorination process and the simultaneous creation of conjugated double bonds in the PVC samples. Interestingly, the untreated PVC experienced swift alterations during aging, eventually turning black. In sharp contrast, the modified PVC film demonstrated improved thermal and color stability, displaying greater resistance to discoloration. High-resolution photographs of modified PVC films were systematically captured as the temperature increased, serving as an additional indicator for the thermal influence on the surface. The modified PVC film reached a certain threshold without a discernible color change, indicative of a significant point of stability even under elevated temperatures. This robustly underscores the efficacy of the introduced modifications in safeguarding

the color stability of PVC films during thermal exposure.

The observed resistance to coloration in the modified PVC film positions it as a promising candidate for applications where sustained color stability under thermal stress is paramount. The combined optical microscopy and high-resolution photography analyses provide a holistic perspective on the color stability dynamics of PVC films, thereby affirming the utility of the Schiff base modifications in mitigating the adverse effects of thermal aging.

#### **4- Conclusions**

The objective of preparing modified PVC to enhance thermal stability and prevent thermal degradation has been successfully realized. The oven-aging test proved instrumental in revealing improvements in the initial color and the onset of blackening time for PVC films. A comprehensive suite of characterization methods, encompassing the analysis of particular functional groups in the infrared spectra, weight loss measurements, and the examination of morphology through optical microscopy, scanning electron microscopy, and atomic force microscopy, was employed to rigorously assess the impact of the introduced modifications. The cohesive findings consistently underscored that the color change process took a more extended duration in the modified PVC. Notably, the modified PVC exhibited a noteworthy enhancement in thermal stability, attributed to its pronounced HCl neutralization ability and the effective passivation of labile chlorine atoms within the polymer chains. These amendments not only elevated the thermal resistance of PVC but also played a pivotal role in preventing thermal degradation. This marks a significant stride toward the development of more

stable and reliable PVC materials, with implications for a spectrum of applications demanding heightened thermal performance and prolonged material lifespan. The positive outcomes from this study affirm the efficacy of the employed modifications and open avenues for further advancements in the realm of thermal stabilization of polymers, especially PVC, crucial in diverse industrial sectors.

### **Acknowledgment**

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