TẠP CHÍ HÓA HỌC & ỨNG DỤNG
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SỐ ĐẶC BIỆT

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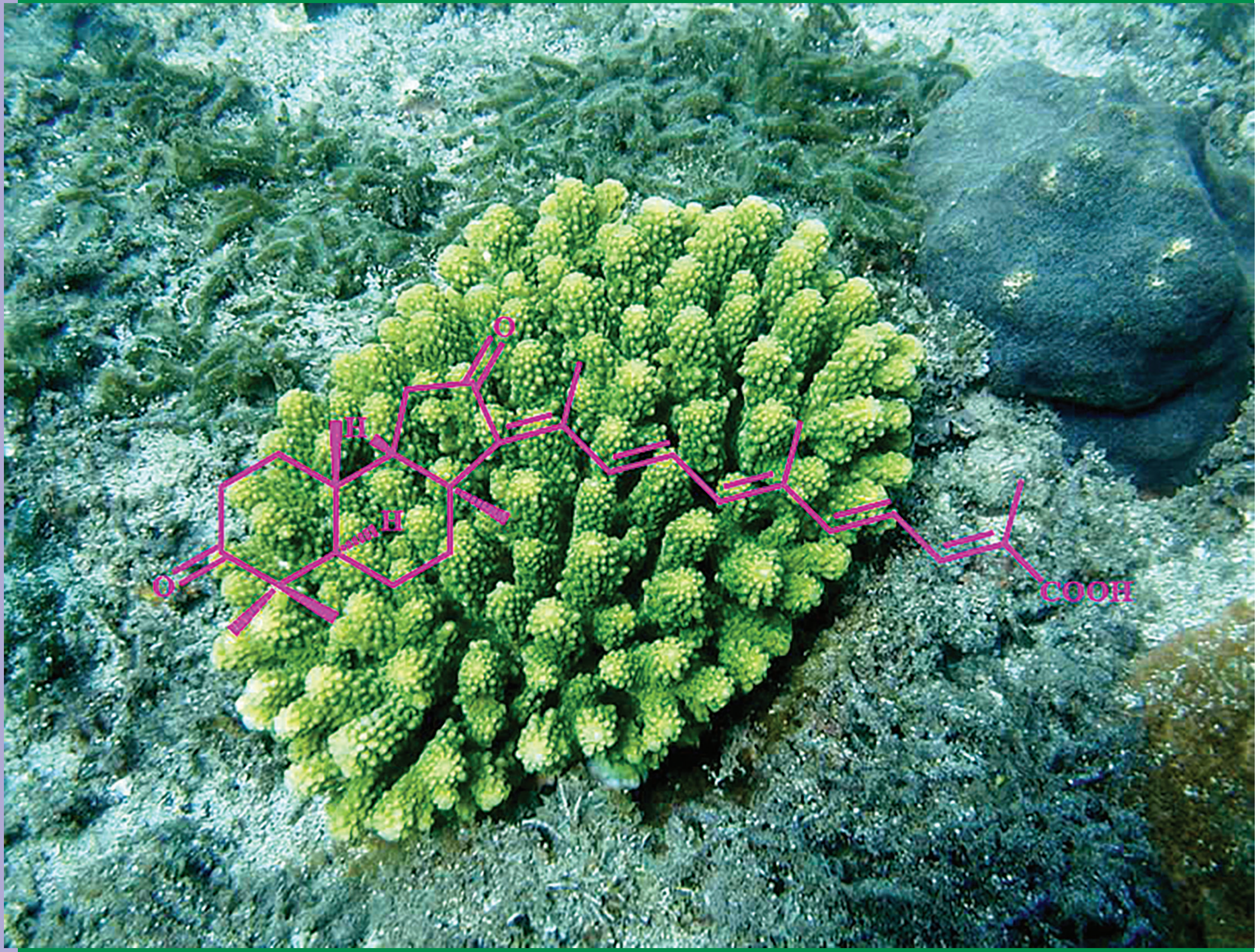

CHARACTERIZATION OF ACRYLIC COATINGS CONTAINING POLY(TRIETHYLAMMONIUM 3-THIOPHENEACETATE) POLYELECTROLYTE AND NANO-SiO$_2$

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SUMMARY:

The fabrication of acrylic coatings containing polythiophene-based conjugated polyelectrolyte and nano-SiO$_2$, using oxidative coupling polymerization to create the water-soluble polyelectrolyte triethylammonium poly(3-thiophene acetate), was successful. The chemical structure, morphology, and mechanical properties of acrylic/polythiophene/nano-SiO$_2$ coatings were characterized by Fourier Transform Infrared (FT-IR) spectroscopy, Field emission scanning electron microscope (FESEM), and measurements including abrasion resistance, gloss, and pencil hardness. The results indicated that adding polythiophene and nano-SiO$_2$ as reinforcing additives improved the mechanical properties of the acrylic coating. The acrylic coating with the nano-SiO$_2$ and polythiophene content of 2wt% and 2wt% achieved optimal properties in gloss, pencil hardness, and abrasion resistance results. The abrasion resistance of the coating was 346.4l/mil, and the gloss was 121.6 GU.

Keywords: *Nano-SiO$_2$, polythiophene, polyelectrolyte, acrylic coating, abrasion resistance.*

I. INTRODUCTION

In recent years, the development of organic paints and coatings has grown significantly due to the rising need for sustainability and aesthetics in buildings. The current problems in this area are the production of coatings with good mechanical properties, including hardness, gloss, weather resistance, and improvement of the material's surface qualities under environmental conditions. Acrylic resin coating, with its high adhesion strength, color stability enhancement, and weather resistance, is extensively employed[1-3]. Research has indicated that adding photo stabilizers, modified nanocomposite particles, or additives such as plasticizers, adhesives, and crosslinkers can improve these characteristics of acrylic coatings [4-6].

Currently, scientists and industry have paid particular attention to nanotechnology[7-9]. Among the used nanoparticles, nano-SiO$_2$ is studied and utilized in numerous high-tech manufacturing industries, such as drug carriers, corrosion inhibitors, antibacterial agents, or nanomaterials to reinforce polymer-based materials[10-13]. Incorporating nano-SiO$_2$ into the coating can provide it with novel, desirable properties while enhancing its mechanical, thermal, and anti-corrosion qualities. Additionally, using silane-grafted nano-SiO$_2$ or polymer-grafted nano-SiO$_2$ can improve the dispersion and compatibility of nano-SiO$_2$ in a polymer matrix[14-18]. For example, the grafting of poly(ethylene oxide) onto nano-silica has demonstrated enhanced dispersibility and adhesion of nano-SiO$_2$ in polyurethane coatings[19]. Upon adding nano-SiO$_2$ modified with some polythiophene to waterborne acrylic coatings, the UV absorption intensity and other mechanical characteristics, including abrasion resistance, pencil hardness, and gloss, were improved[14]. This research highlights that using nano-silica and polythiophenes as additives can enhance the characteristics of acrylic coatings. In particular, water-soluble polythiophene derivatives or polyelectrolyte polythiophene are of special significance among CPEs due to a unique combination of good environmental stability, conductivity, and versatility of substituted π-conjugated backbone in various technical applications[20-22].

In our studies, nano-SiO_2 and polyelectrolyte polythiophene as reinforcing additives were added to acrylic coatings, aiming to enhance the mechanical characteristics of coatings. Polyelectrolyte polythiophene was synthesized from poly(3-thiophene acetic acid) using an oxidative coupling polymerization reaction with $FeCl_3$ catalyst. The influence of nano-SiO_2 content on the mechanical characteristics of acrylic/polythiophene/nano-SiO_2 coatings, including gloss, pencil hardness, and abrasion resistance of coatings, were investigated.

II. EXPERIMENTAL

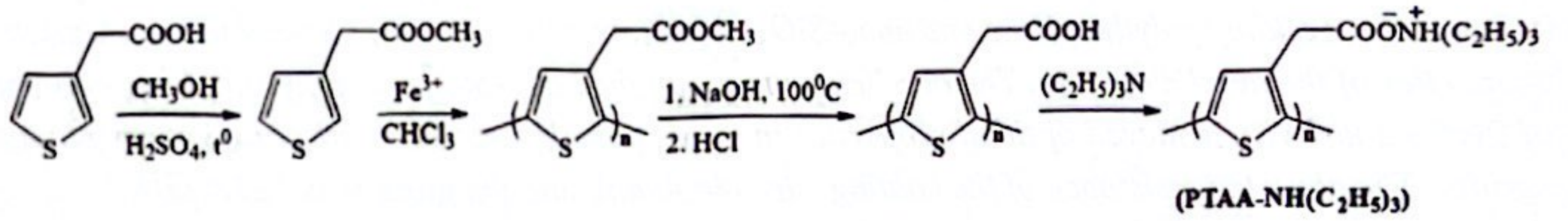
2.1. Chemicals

2-(Thiophen-3-yl)acetic acid, anhydrous iron (III) chloride (98%), triethylamine, sulfuric acid (98%), hydrochloric acid (37%), sodium hydroxide, methanol, and chloroform (all obtained from Merck), and nano-SiO_2 (from rice husk ash, ~50-200nm) were directly used as purchased.

Acrylic emulsion resin Plextol R 4152, which has a total solids content of 50%, pH of 7-8.5, a viscosity of 500-3,000cps, and particle size of 0.13 μm, was purchased from Synthomer Company, USA.

2.2. Fabrication of acrylic R4152 coatings containing poly(triethylammonium 3-thiopheneacetate) polyelectrolyte and nano-SiO_2

2.2.1. Synthesis of poly(triethylammonium 3-thiopheneacetate) polyelectrolyte PTAA-NH(C_2H_5)$_3$



Scheme 1. The synthesis of poly(triethylammonium 3-thiopheneacetate) polyelectrolyte

The synthesis of poly(triethylammonium 3-thiopheneacetate) polyelectrolyte (PTAA-NH(C_2H_5)$_3$) was reported in our previous study[23].

2.2.2. Fabrication of acrylic coatings containing PTAA-NH(C_2H_5)$_3$ and nano-SiO_2

0.1g of PTAA-NH(C_2H_5)$_3$ was dissolved in 1ml of deionized water by TPC-15H Ultrasonic Bath Equipment with a capacity of 450W and a vibration frequency of 20kHz. This step was carried out for 120 minutes to ensure good dispersion of polyelectrolyte particles in water. After that, nano-SiO_2 and 10g of acrylic 4,152 resin were added to the mixture. The newly obtained mixture was further ultrasonically vibrated for 120 minutes and magnetic stirred for 2 hours with an IKA RW16 stirrer at 400rpm speed to disperse the nanoparticles evenly in the acrylic resin mixture. The mass compositions of the acrylic/polythiophene/nano-SiO_2 coatings (APS0.5, APS1, APS2, and APS4) are displayed in Table 1.

Table 1: Composition of acrylic coatings containing PTAA-NH(C_2H_5)$_3$ and nano-SiO_2

No.	Sample symbol	SiO_2 (g)	PTAA-NH(C_2H_5)$_3$ (g)	H_2O (ml)	Acrylic R4152 (g)
1	A0	-	-	1.0	10
2	AP0	-	0.1	1.0	10
3	APS0.5	0.025	0.1	1.0	10
4	APS1	0.050	0.1	1.0	10
5	APS2	0.100	0.1	1.0	10
6	APS4	0.200	0.1	1.0	10

Coating A0 and AP0 were prepared using the same method to serve as a control sample.

The samples were created with a wet film thickness of 120μm using an Erichsen Film Applicator (model 360) on glass and concrete (equivalent to a dry film thickness of 30 ± 2μm). Before determining the characteristics and properties of the coating, the coating samples were allowed to dry for seven days under standard conditions. They were kept at standard temperatures of 25°C and 60% relative humidity for 24 hours before testing.

2.3. Devices and methods

FT-IR spectroscopy

FT-IR spectroscopy was investigated using a Nexus 670 ThermoNicolet Fourier Transform Infrared Spectrometer from Nicolet. The spectrum was scanned in the 4,000–400cm^{-1} range with a resolution of 8cm^{-1}, and the number of scans was 32 times.

Thermal stability

Thermo-gravimetry experiments were performed using a TGA-50 Shimadzu Thermal Analysis Instruments (Japan). The samples were heated from room temperature to 600°C at a rate of 10°C/min with a flow rate of 50cm^3/min.

Morphology

Structural morphology of the coatings was analyzed in a Hitachi S-4800 SEM instrument (Japan).

Abrasion resistance

Following ASTM D968-15 standard, the Falling Sand Abrasion TesterTM was used to evaluate the abrasion resistance of the coating by measuring the surface degradation when the paint film was under the falling sand force at a predefined height of 0.914 meters. Furthermore, the paint film thickness was deducted as the mean from 3 measures using the ElektroPhysik Minitest 600.

Gloss measurement

Erichsen Picogloss (model 503) was used to test the gloss at 60° angle of the formula coatings after an accelerated aging process according to ISO 2813:2014. The ratio of the aged coating's gloss to that of the original coating was used to calculate the gloss retention.

Pencil hardness

This technique assessed the hardness and scratch resistance of the film according to the ASTM D 3363 standard. The graphite scale ranged from 6B to 8H, the pencil tip was subjected to 500g of pressure, and the angle between the pencil and measuring surface was 45 degrees.

III. RESULTS AND DISCUSSION

3.1. Morphology of acrylic coatings containing PTAA-NH(C_2H_5)$_3$ and nano-SiO$_2$

The results show a relatively even distribution of the polythiophene and nano-SiO$_2$ particles throughout the polymer matrix (Figure 1). When adding polythiophene coating with 0.5wt%, 1 wt%, and 2wt% nano-SiO$_2$ (APS0.5, APS1, and APS2), all paint films have identical morphology. The nanoparticles are well-dispersed in the polymer matrix, with only a few small clumps. However, when 4wt% of nano-SiO$_2$ particles are added to the APS4 coating, the SiO$_2$ nanoparticles tend to aggregate considerably, forming particle clusters that are tens of nm in size and leading to defective areas in the paint films.

Therefore, it is clear that the ideal outcomes are obtained with 1-2 wt% nano-SiO$_2$ particles and 2wt% polythiophene polyelectrolyte particles distributed throughout the acrylic resin system.

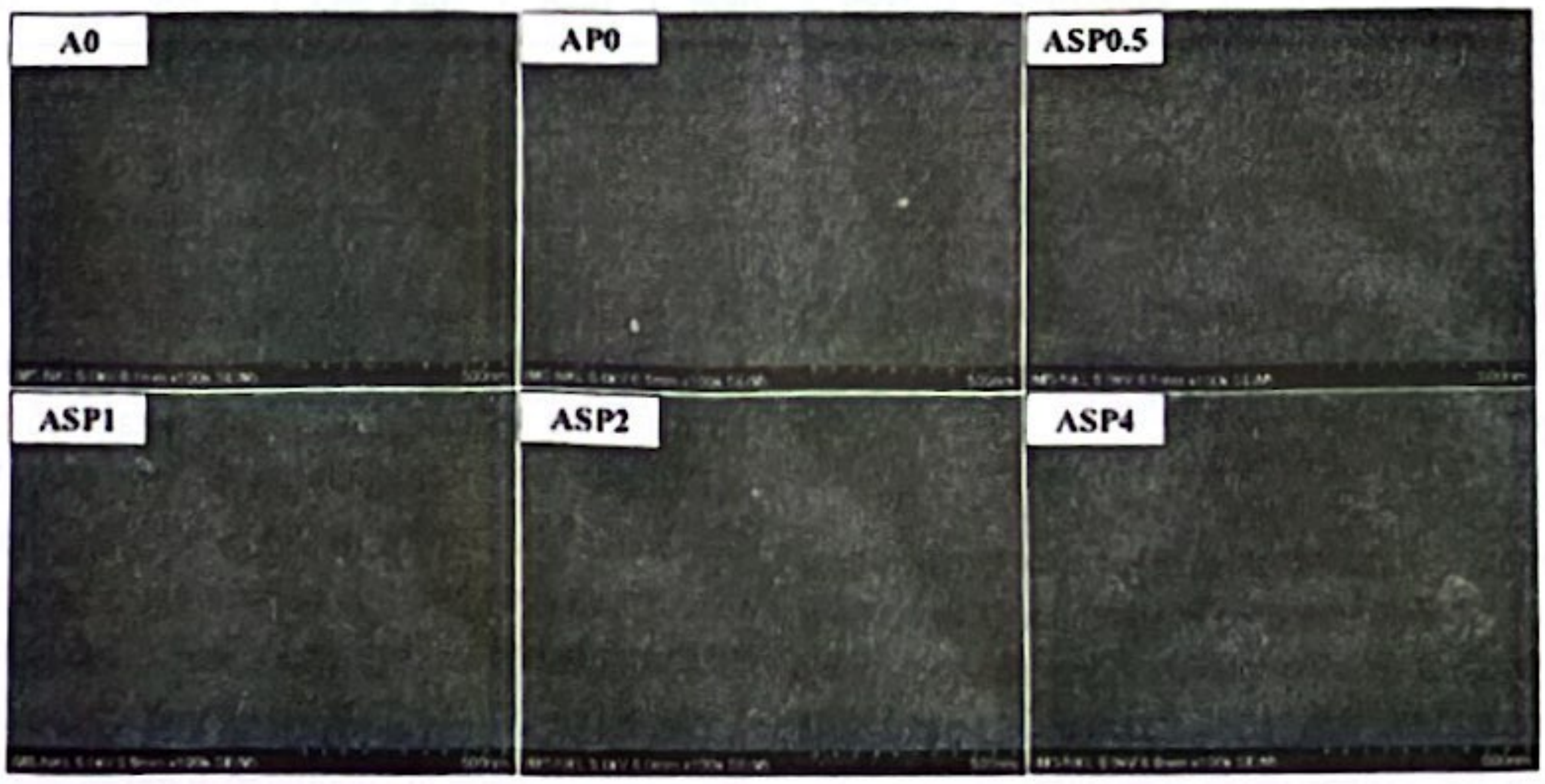


Figure 1. FE-SEM images of acrylic/ poly(triethylammonium 3-thiopheneacetate) polyelectrolyte/nano-SiO$_2$ coatings

3.2. FT-IR spectra of acrylic coatings containing PTAA-NH(C_2H_5)$_3$ and nano-SiO_2

Based on the IR spectra of all acrylic/poly(triethylammonium 3-thiopheneacetate) polyelectrolyte/nano-SiO_2 coatings (Figure 2) and some major vibrations of acrylic/polythiophene/nano-SiO_2 coatings (Table 2), it can be seen that the absorption spectra at 1,098cm^{-1} and 808cm^{-1} corresponding to the asymmetric and symmetric vibrations of the Si–O group in nano-SiO_2, respectively[24]. In the coating containing polyelectrolyte and acrylate systems, there are characteristic peaks for the vibration of the –C=O ester group at around 1,745–1,722cm^{-1} and the vibration of the –CH sp^3 group at around 2,946cm^{-1}. In addition, a strong blunt band at around 3,500cm^{-1} characterizes the valence vibrations of –OH bonds of hydroxyl groups on the SiO_2 surface and –NH bond of the side group in polyelectrolyte. The characteristic band for the deformation vibration of the C–S bond of the thiophene ring is covered by the characteristic vibrational region of acrylate. The bands in the 1,400-1,100cm^{-1} region of the bending mode of the C–C–O and C–C=O bonds belong to esters in the backbone chain[25].

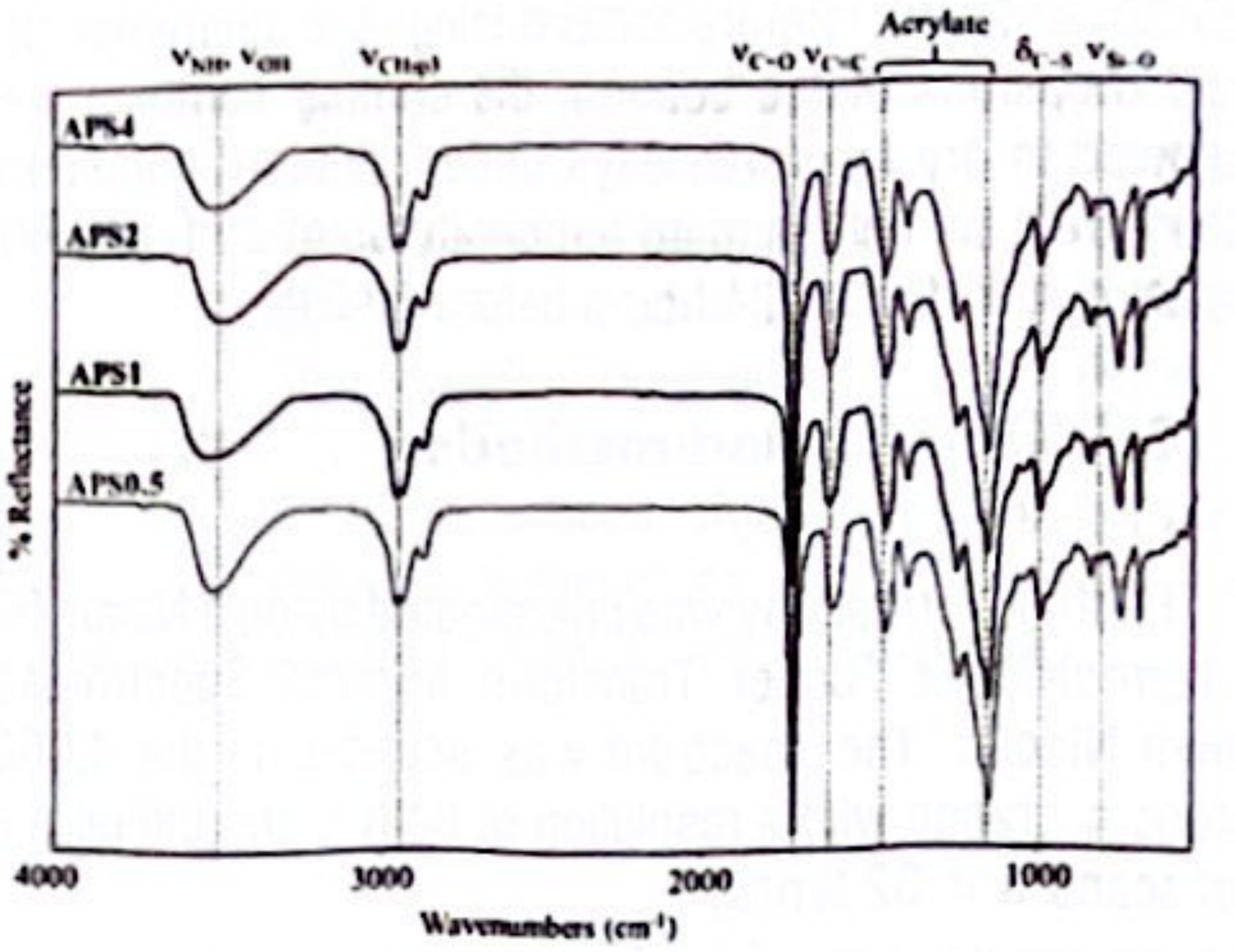


Figure 2. FT-IR spectra of acrylic/ poly(triethylammonium 3-thiopheneacetate) polyelectrolyte/nano-SiO_2 coatings

Theoretically, the distinctive spectral bands for acrylate group bonds at 1,635cm^{-1} and 982cm^{-1} will almost disappear during the production of the acrylic/poly(triethylammonium 3-thiopheneacetate) polyelectrolyte/nano-SiO_2 paint system, indicating that these groups have taken part in the bonding process. The spectral bands at this point, however, continue to occur because they represent the deformation vibration of the C–S bond in polyelectrolyte PTAA-NH(C_2H_5)$_3$ and the vibration of the conjugated system C=C–C=C, respectively.

Table 2: Some major vibrations in IR spectrum (cm^{-1}) of acrylic/poly(triethylammonium 3-thiopheneacetate) polyelectrolyte/nano-SiO_2 coatings

Coating	ν_{N-H}, ν_{O-H}	$\nu_{-CH\ sp^3}$	$\nu_{C=O}$	$\nu_{C=C}$	ν_{C-C}	δ_{C-S}	ν_{Si-O}	Acrylate
APS0.5	3511	2949-2922	1728	1604	1448	1040; 989	809	1400-1100
APS1	3509	2952-2922	1729	1608	1448	1043; 983	809	1400-1100
APS2	3496	2946-2922	1722	1610	1445	985	810	1400-1100
APS4	3515	2952-2925	1745	1604	1445	986	809	1400-1100

3.3. Mechanical properties of of acrylic coatings containing PTAA-NH(C_2H_5)$_3$ and nano-SiO_2

3.3.1. The gloss

Gloss is one of the necessary properties of the paint film surface. Figure 3 illustrates how the content of nano-SiO_2 affects the gloss of acrylic/polythiophene polyelectrolyte coatings. The control film A0 without SiO_2 and PTAA-NH(C_2H_5)$_3$ has the lowest gloss measured at 92.2 GU. The gloss of the paint film is enhanced with the presence of PTAA-NH(C_2H_5)$_3$ (AP0), or both PTAA-NH(C_2H_5)$_3$ and nano-SiO_2 (APS0.5, APS1, APS2 and APS4). The paint film with the highest gloss result, measured at 121.6 GU, is APS0.5, which contains 2wt% nano-SiO_2. When nano-SiO_2 particles are in the 0.5–1 wt% range, they can fill the defect area and cover the surface of the coating. However, as the concentration of nano-SiO_2 particles increases to 4wt%, more surface flaws develop because of the excess nano-SiO_2, leading the decrease in the gloss of the coating in the sample APS4.

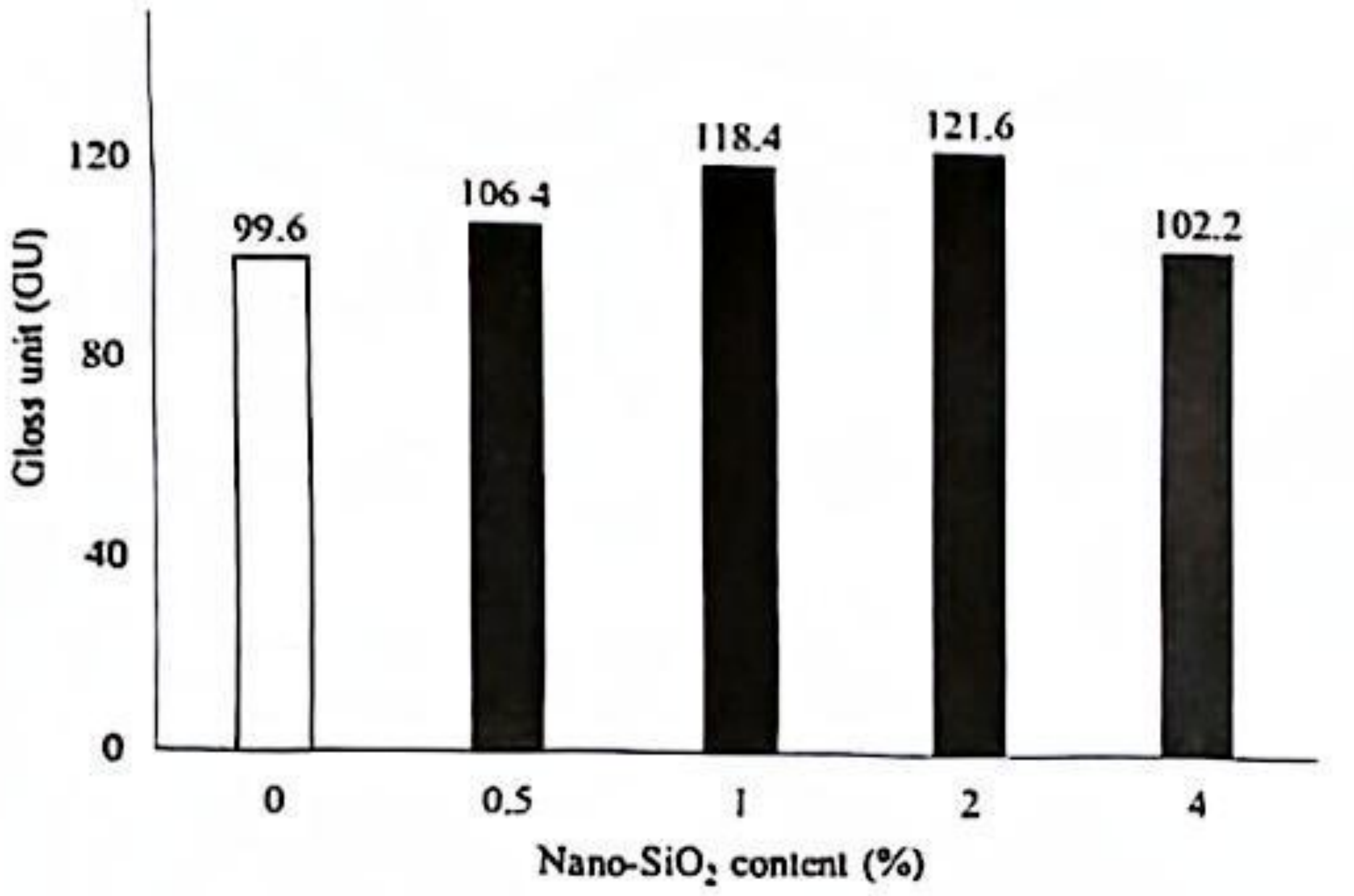


Figure 3. Influence of nano-SiO_2 content on the gloss of acrylic/poly(triethylammonium 3-thiopheneacetate) polyelectrolyte coatings

3.3.2. Abrasion resistance and pencil hardness

The influence of nano-SiO_2 content on the abrasion resistance and pencil hardness of acrylic/polythiophene coatings are presented in Figure 4 and Table 3.

Firstly, the mechanical properties of the coating are improved by the presence of nano-SiO_2 particles, and the results of pencil hardness and abrasion resistance are proportionate. These results reveal that the ideal relative hardness and pencil hardness values, or 346.4l/mil and 2H, respectively, are found in the APS2 sample that contains 2wt% nano-SiO_2, whereas the control sample A0 exhibits the lower values of 91.4l/mil and HB.

Secondly, the nano-SiO_2 content added to the coating in the range of 0.5wt% to 2wt% is proportionate to the abrasion resistance and pencil hardness values. Nevertheless, the abrasion resistance value of the APS4 sample drops to a value that is even lower than that of the APS0.5 sample, which contains 0.5% nano-SiO_2 content, when the silica concentration grows to 4wt%. This is because an excessive concentration of nano-SiO_2 can cause the agglomeration, resulting in uneven distribution of the particles throughout the sample and reducing the compactness of the material.

Third, the results of abrasion resistance and pencil hardness values of the coatings are also consistent with the FE-SEM image (Figure 1) and gloss (Figure 3) results.

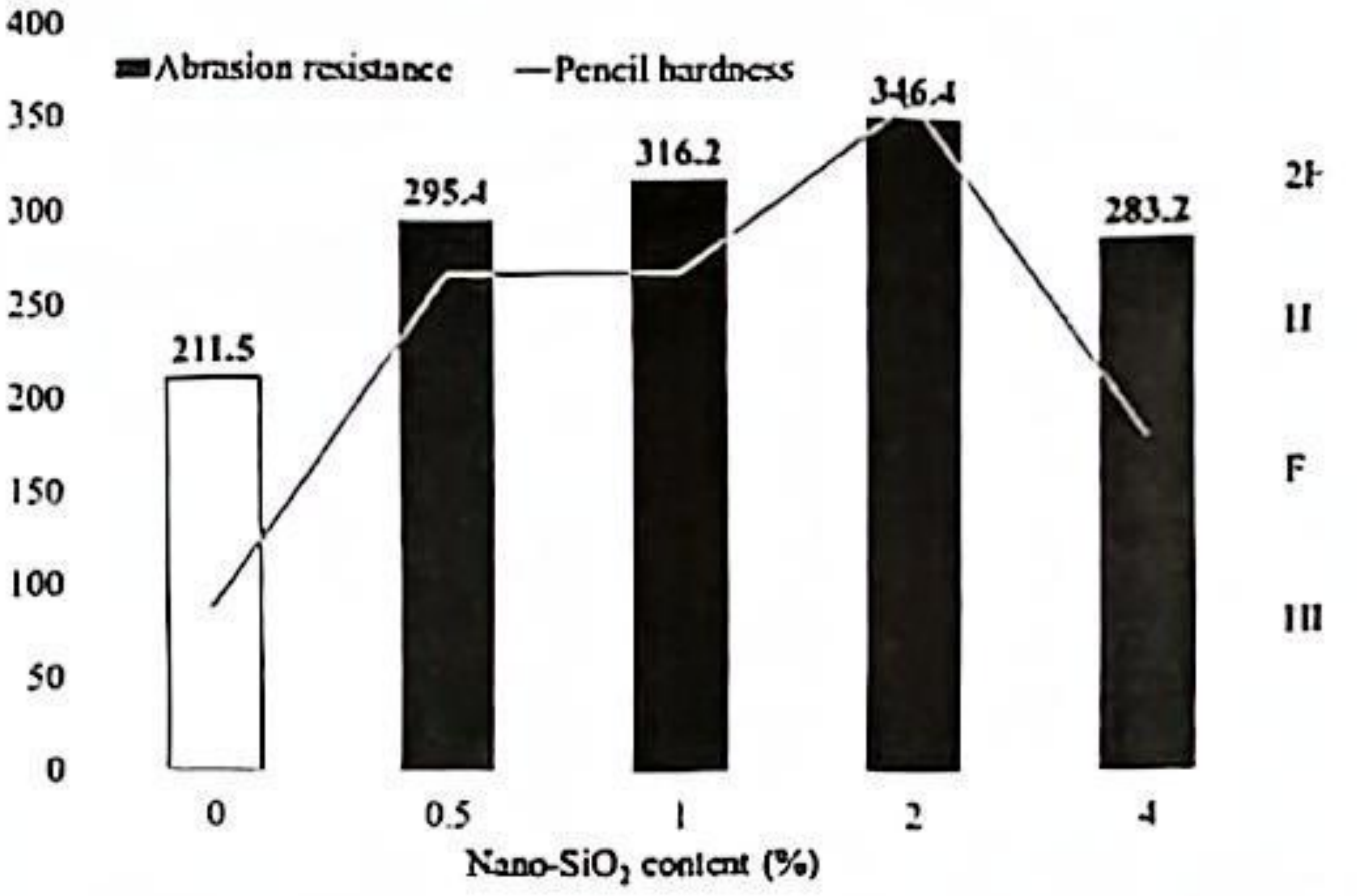


Figure 4. Influence of nano-SiO_2 content on the abrasion resistance and pencil hardness of acrylic/poly(triethylammonium 3-thiopheneacetate) polyelectrolyte coatings

Table 3: Effect of nano-SiO_2 on mechanical properties of acrylic/poly(triethylammonium 3-thiopheneacetate) polyelectrolyte coatings

No.	Mechanical characteristics	Content of nano-SiO_2, %				
		0	0.5	1	2	4
1	The gloss, GU	99.6	106.4	118.4	121.6	102.2
2	Pencil hardness	HB	H	H	2H	F
3	Abrasion resistance, L/mil	211.5	295.4	316.2	346.4	283.2

IV. CONCLUSION

Poly(triethylammonium 3-thiopheneacetate) polyelectrolyte (PTAA-$NH(C_2H_5)_3$) was synthesized from poly(3-thiophene acetic acid) using oxidative coupling polymerization reaction with $FeCl_3$ catalyst. Based on the highest water solubility, the polyelectrolyte was used as a reinforcing additive to improve the properties of acrylic coatings. Furthermore, the impact of nano-SiO_2 content on the mechanical properties of acrylic coatings that incorporate PTAA-$NH(C_2H_5)_3$ was investigated. The dispersion of polythiophene polyelectrolyte and nano-SiO_2 particles in the acrylic resin matrix was satisfactory, with 2wt% of each material for optimal outcomes. In addition, the presence of SiO_2 nanoparticles and polyelectrolyte polythiophene significantly improved the morphology, gloss, pencil hardness, and abrasion resistance of the acrylic coatings, with the best results achieved from the sample containing 2wt% both SiO_2 nanoparticles and polythiophene. In particular, at this content of poly(triethylammonium

3-thiopheneacetate) polyelectrolyte and nano-SiO$_2$, the abrasion resistance and gloss of the coating APS2, respectively, were 346.4l/mil and 121.6 GU.

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