

Synthesis and evaluation of surfactants with functional derivatives as additives for improvement of the mechanical and thermal properties of drinking water pipes

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Abstract.

In the present study, some thiol surfactants were prepared. Polyester / (nano bentonite) nano composite (A), polyester/(5% nano Bentonite) / thiol surfactant (I) nano composite (B), polyester / (5% nano Bentonite)/thiol surfactant (II) nano composite (C) and Polyester/(5% nano Bentonite)/thiol surfactant (III) nano composite D were fabricated. The mechanical, thermal properties and morphological structure of fabricated composites were studied. The results showed that nano Bentonite powder / polyester composite improve these properties at 5% weight and begins to weaken as the filling ratios increased. However using thiol surfactants as additives to polyester / (nano bentonite) nano composites improve the thermal and mechanical properties of nano composites. But nano composite (D) gave the optimum thermal and mechanical properties which can be used in manufacture of drinking water pipes.

Keywords: Nano composite; surfactant, mechanical and thermal properties ,drinking water pipes.

1. Introduction

Polyester resins are one of the most important matrix materials which can be used in the manufacture of glass fiber reinforced composites [Zaske, O. C., Goodman, H. S., 1998]. For many years, polyester composites have been used in very varied technologies, like naval constructions, off-shore applications, water pipes, building construction, automotive applications, etc. [Baudry, A. J., Dufay, N. R., et al., 1998], [Rebeiz, K. S., 1996], [Peter, J. S., Andrew, J. P., et al., 2006], [Peter, J. S., Michael, S. J., et al., 2006], [Ayman, M. A., Ashraf, M. E., et al., 2007] and [Lin, S. P., Shen, J. H., et al., 2008]. Polyester resins exhibit several useful characteristics due to the high degree of cross-linking between individual polymer chains [Matthews, F. L. Rawlings, R. D., 1999].

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Polymer materials have many of benefits such as ease of production, lightweight and often ductile nature so there can be used in industry. However, they have some disadvantages, such as low modulus and strength compared to metals and ceramics. We can improve their properties to overcome these problems so natural materials can used such as clay, chrysotile and lingo cellulosic fibers as filler in fabrication of polymer nano composites [Varlot K., Reynaud, E., et al.,2001] and [Almansoori, A., Seabright, R., et al., 2017].

Nano composites are considered as the next industrial revolution materials which have a high performance material exhibit unusual property combinations. Applications of nano composites offer new technology and business opportunities for several sectors of the plastics, aerospace, automotive, electronics and biotechnology industries[Satyanarayana, K.G., Camargo, P. H.C., et al.,2009] and[Abulyazied, D. E. , Mokhtar ,S. M. et al.,2014].

Polymer nano composites are materials consisting of addition of particals at nano-scale which disperse in polymer to give a significant improvement of properties when compared to the base polymer[Lanciano, G.; Greco, A.; Maffezzoli, A.,et al., 2009], [Greco, A., Esposito Corcione, C., et al.,2010], [Corcione,C.E., Cavallo, A. , et al.,2011], [Calò, E., Massaro, C., Terzi,R., et al.,2012] and [Greco, A., Maffezzoli, A., et al.,2012]. Both thermoplastic as well as thermoset based clay nano composites have been widely studied, showing that in most cases improved the properties [Indennidate, L., Cannoletta, D ., et al.,2010], [Greco, A., Corcione, C.E., et al.,2010] and [Corcione, C.E., Mensitieri, G. et al. , 2009]. For this reason, academic research focuses on alternative materials for the production of polymer nano composites. These include addition of organic fillers [Frigione, M., Calò, E., J. ,2008], [Corcione ,C.E., Frigione, M., 2009] and [Di Lorenzo, M.L., Frigione, M., 1997]or use of different types of inorganic nano fillers such as clays have gained interest due to their exceptional properties like higher mechanical strength and thermal resistance of polymeric materials. These properties could further be improved by incorporating a certain amount of clay in the polymeric materials [Martone, A., Grassia, L., et al.,2012], [Monti, M., Natali, M., Torre, L. et al.,2012], [Monti, M., Natali M., et al.,2011] and[Terenzi, A., Vedova, C., et al.,2008].

In this work, we use surfactants with functional derivatives as additive of nano composites for improvement of the mechanical and thermal properties of drinking water pipes. So we study the behavior of pure resin (polyester), nano bentonite (clay) and organo clay powders. Static tensile, compressive and flexural tests are first carried out on polyester and (nano bentonite (clay) and organo clay)/polyester composites to evaluate the mechanical properties. Also the thermal gravimetric analyses (TGA) carried out on all fabricated nano composites to evaluate their thermal properties.

2. Experimental

2.1 Materials and Chemicals

2.1.1 Materials

The resins used in this study was o-phthalic polyester resin obtained from Arab Company for Developed Materials of Egypt, it with medium viscosity as supplied by the manufacture. In addition, Bentonite was obtained from Beni suef /Egypt region. The raw bentonite with elemental composition contained montmorillonite as major element was used as filler material.

2.1.2 Chemicals

Chemicals used in this study and their specifications were illustrated in **Table 1**.

Table 1. Chemicals used in this study

Compound	Source	Purity
2-Mercapto pyridine	Aldrich	99%
1-Bromo decane	Riedel-deHaen	97%
1-Bromo dodecane	Riedel-deHaen	97%
1-Bromo hexadecane	Riedel-deHaen	97%
Acetone	Bio.chem	99%
Benzene	Adwic	99.5%
Petroleum ether	Adwic	60-80%

2.2 Preparation

2.2.1 Synthesis of heterocyclic surfactants (cationic thiol surfactants I,II and III) [Azzam, E. M. S., Sami, R. M., et al.,2012]

A mixture of alkyl halides with different alkyl chain lengths of (0.02 mol,4.42 gm) of 1-bromo decane, (0.02 mol,4.98 gm) of 1- bromo dodecane and (0.02 mol,6.1 gm) of 1- bromo hexadecane and (0.02 mol, 2.22 gm) of 2-mercapto pyridine in acetone (20ml) was refluxed at 40°C for 5 hour. The reaction mixture was concentrated to evaporate acetone. The obtained precipitate was crystallized using mixture of benzene and petroleum ether 60-80% , then dried to obtain cationic thiol surfactants 1-decane,dodecane and hexadecane 2-mercapto pyridine-1-ium bromide (I,II and III) respectively as shown in **Fig.1**.

Where

Cationic surfactant (I) with yellow crystals, m.p. 92°C, yield 87.3 %, M.F. C₁₅H₂₆NSBr,
 Cationic surfactant (II) with pale yellow crystals, m.p. 96°C, yield 90.4 %, M.F. C₁₇H₃₀ NSBr,
 Cationic surfactant (III) with white crystals , m.p. 94°C, yield 88.8 %, M.F. C₂₁H₃₈ NSBr.

2.2.2 Preparation of Nano Bentonite

Nano-particles of bentonite have been prepared by nano-grinding bentonite as crushed to finer particles and sundried for 5 days to ease pulverizing and sieving. Then was ground to powder with the aid of mortar and pastle, and then sieved with a rota shaker to obtain 63 μ fractions to Suit API specification for local bentonite [Abdou, M.I., El-Sayed Ahmaed, H., 2011] and [Sharma,U. A. Suri, M. Mukul, 2004] Nano-bentonite was prepared by crushing the local bentonite with the aid of a planetary ball mill PM 400 .The chemical analysis of bentonite powder and Nano bentonite powder as shown in Table 2.

Table 2: Chemical Analysis of Bentonite powder and Nano Bentonite Powder

Material	Bentonite %	Nano bentonite %
LOI*	13.40	10.20
Na ₂ O	1.46	2.37
MgO	1.39	1.27
Al ₂ O ₃	17.60	16.10
SiO ₂	42.10	48.30
P ₂ O ₅	0.25	0.18
SO ₃	3.41	3.59
Cl-	1.21	0.31
K ₂ O	0.70	1.18
CaO	6.79	5.94
TiO ₂	0.92	1.20
MnO	0.10	0.10
Fe ₂ O ₃	10.61	9.16
TOTAL	99.94	99.90

* loss of ignition

2.3 Fabrication of nanocomposite

Firstly, the drying conditions of filler is known to play a crucial effect on the process results, since nano bentonite is hydrophilic and the moisture can cause significant decrease in the thermal stability of nanobentonite and as a result decrease the synergistic properties between the polyester and nano bentonite. Thus, nano bentonite was dried at 80°C before used in fabricating with polyester. Addition of cationic thiol surfactants (I,II and III) to nano bentonite give organo clay (I,II and III) respectively.

A variety of fabricating nano composites specimens were prepared according to **Table 3**. All fabricated nano composites were then left for 24 hour at room temperature, then specimens can be removal and the test specimens of required sizes were cut out from the sample sheet.

Table 3: Fabrication of nano composite specimens

Group No	Material	Nano Bentonite %
1	Polyester resin	0
2	(Polyester/ Nano Bentonite) Nano composite (A)	2 5 10
3	(Polyester/ (Nano Bentonite Thiol surfactant I) Nano composite (B)	5
4	(Polyester/ Nano Bentonite Thiol surfactant II) Nano composite (C)	5
5	(Polyester/ Nano Bentonite Thiol surfactant III) Nano composite (D)	5

2.4 Testing

2.4.1 Mechanical Properties

The prepared test specimens were subjected to static tensile, compression and flexural tests. All tests were performed at room temperature of 23°C. In each test, five samples were tested and the average value was tabulated were carried out using 100 KN computerized AG-Shimadzu-Autograph Universal Testing Machine at testing speed was 1 mm /min.

Tensile testing samples were prepared in dumbbell shapes with dimensions as per [ASTM D638-14]. The samples were loaded in tension at a cross-head speed of 1mm/min to determine their tensile behavior. A three point flexural test was carried out to determine the inter laminar shear strength (ILSS) of the samples as per [ASTM D790-17].

The slow flexural test specimen has a rectangular cross-section, 9 mm width and thickness 4 mm and 100 mm length. Compressive test was carried out using testing machine at load rate of approximately 0.2 N/mm²S. The specimens were taken according to the standard specification [ASTM D695 – 15] . The compression test specimen has a height equal to width and a width 20mm.

2.4.2 Thermal properties analysis

The thermal characteristics of the polyester/filler fabrication nano composites were studied using thermal gravimetric analysis (TGA) (Shimadzu TGA-50). TGA was used to investigate thermal decomposition behavior of the nano composite fabrication.

2.4.3 Morphology analysis

The morphology of fabricated nano composites was studied using Inspect s model scanning electron microscope (SEM). SEM also used for studied the dispersion of filler particles in the fabrication of nano composites

3. Results and Discussion

In this study, a mixture of thiol surfactants based on 2-mercaptopyridine with alkyl halides with different alkyl chain lengths (I,II and III) were prepared according to the method in experimental part as illustrated in **Fig. 1**.

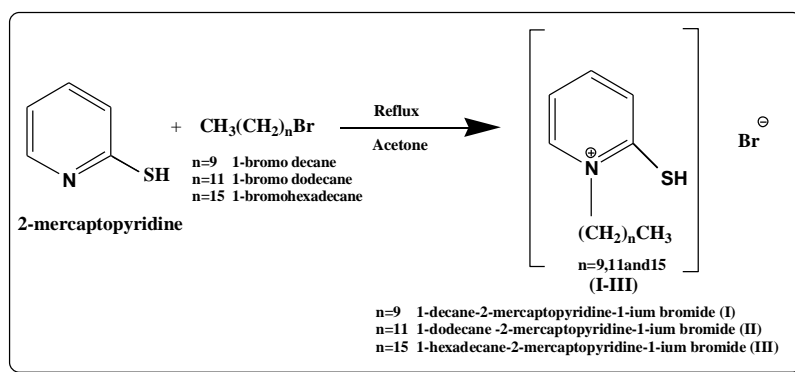


Fig. 1 Synthesis of the cationic thiol surfactants (I,IIandIII)

A prepared thiol surfactants used as additive in the fabrication of polyester nanocomposites to evaluate their mechanical and thermal properties to be applied in the manufacture of drinking water pipes.

3.1 Mechanical Properties

The mechanical properties of all fabrication nano composites are shown in **Table 4**.

Tensile strength (TS) increased with increasing amount of nano bentonite at 2% weight, 5% weight and 10% weight about 2.5 %, 10% and 1.6% respectively compared with polyester resin which showed maximum at 5% weight, so we using thiol surfactants as additives to nano composites at 5 % weight. The tensile strength increased for 5% weight of (organo clay I, organo clay II and organo clay III) compared with polyester resin. The tensile strength of polyester base matrix was improved by about 11 %, 11.7% and 12.5 % respectively

Flexural strength (FS) increased with increasing amount of nano bentonite at 2% weight, 5% weight and 10% weight about 3.3 %, 11.94 % and 6.5% respectively compared with polyester resin which showed maximum at 5% weight, so we used thiol surfactants as additives to nano composites at 5 % weight. The flexural strength increased for 5% weight of (organo clay

I, organo clay II and organo clay III) compared with polyester resin. The improvement of the flexural strength of polyester base matrix was about 12.23 %, 12.66% and 12.94 % respectively.

Also the same trend was clear for compressive strength (CS) which increased at 2% weight, 5% weight and 10% weight about 8.5 % , 20 % and 6% respectively compared with polyester resin which showed maximum at 5% weight, so we used thiol surfactants as additives to nano composites at 5% weight. The compressive strength increased for 5% weight of (nano bentonite, organo clay I, organo clay II and organo clay III) compared with polyester resin. The improvement of the compressive strength of polyester base matrix was about 20.14 %, 20.28% and 20.42 % respectively.

It was observed that, the mechanical properties were optimized at 5 % weight organo clay III content.

Table 4: Mechanical Properties of Fabrication of Nano composites

Material	Nano bentonite (wt.%)	Tensile Strength (MPa)	Flexural Strength (MPa)	Compressive Strength (MPa)
Polyester	0%	60	695	140
(Polyester/ Nano Bentonite) Nano Composites (A)	2%	61.5	718	152
	5%	66	778	168
	10%	61	740	149
(Polyester / Nano Bentonite/ Thiol surfactant I) Nano Composites (B)	5%	66.5	780	168.2
(Polyester / Nano Bentonite)/ Thiol surfactant II) Nano Composites (C)	5%	67	783	168.4
(Polyester / Nano Bentonite/ Thiol surfactant III) Nano Composites (D)	5%	67.5	785	168.6

3.2. Thermal gravimetric analysis (TGA)

The thermo gravimetric analysis showed the weight – loss of the nano composites at different weight (0%, 2%, 5%, and 10%) of nano bentonite as shown in **Fig.2**.

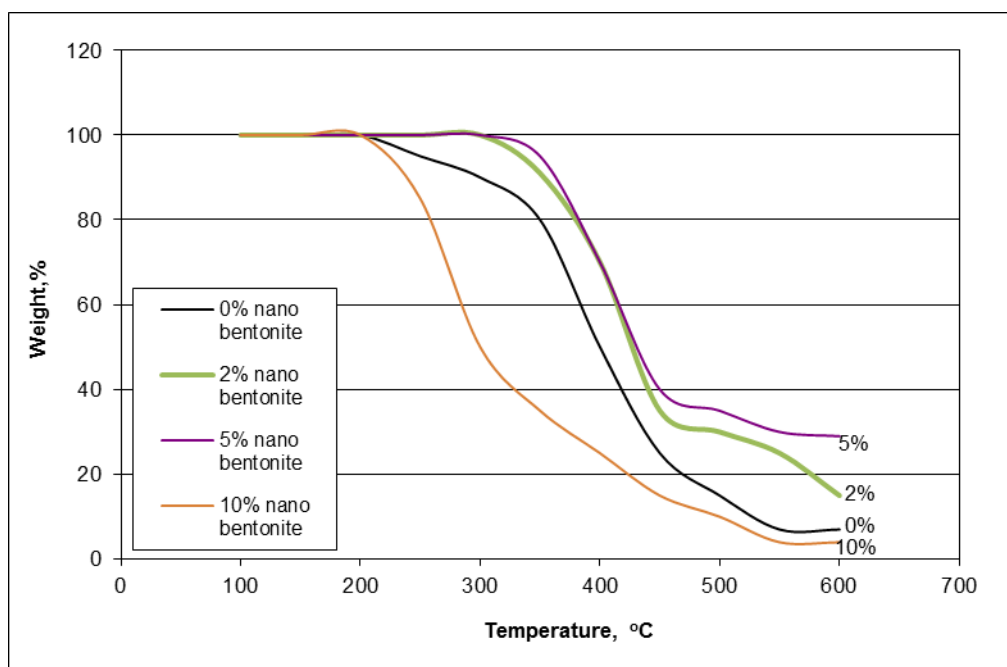


Fig.2 TGA of nano bentonite/ polyester nano composites

TGA curves showed the weight loss of pure polyester show no change up to 200 °C , after 200°C start weight loss change and then decomposition occur at 400 °C to weight loss 50% of original weight of polyester. It is clear that the decomposition temperature of the nano composite shifted towards higher temperatures by loading 2% of weight of nano bentonite, but loading 5 % weight of nano bentonite shift to more higher temperature which indicating higher thermal stability of the nano composite due to high level of dispersion and improve thermal properties ,but after increasing up to 10% weight of nano bentonite shift towards lower temperature i.e. lower thermal stability due to high nano bentonite loading than 5 % weight sample exhibits a structure having large nano bentonite aggregates, which act as stress concentrators.

It was observed that decomposition temperature of nano composite shifted towards higher temperature indicating higher thermal stability of nano composite up to 5% nano bentonite at 450°C, which lower 50°C than pure polyester. So using thiol surfactant as additives to nano composites at 5 % weight.

The thermal gravimetric analyses (TGA) of adding (nano bentonite, organo clay I , organo clay II and organo clay III) show increasing in decomposition temperature and weight loss constant up to 300 °C when compared with pure polyester resin as shown in **Fig. 3**.

TGA curve showed adding the three thiol surfactants (I, II and III) to nano bentonite surface (organo clay I, organo clay II and organo clay III) respectively show increasing in decomposition temperature than nano bentonite only, which improved of thermal stability of nano composites.

It is clear that no significant change between two curves when adding thiol surfactants (I and II) to nano bentonite (organo clay I and II), but adding thiol surfactant III to nano bentonite (organo clay III) show higher thermal stability than (organo clay I and organo clay II).

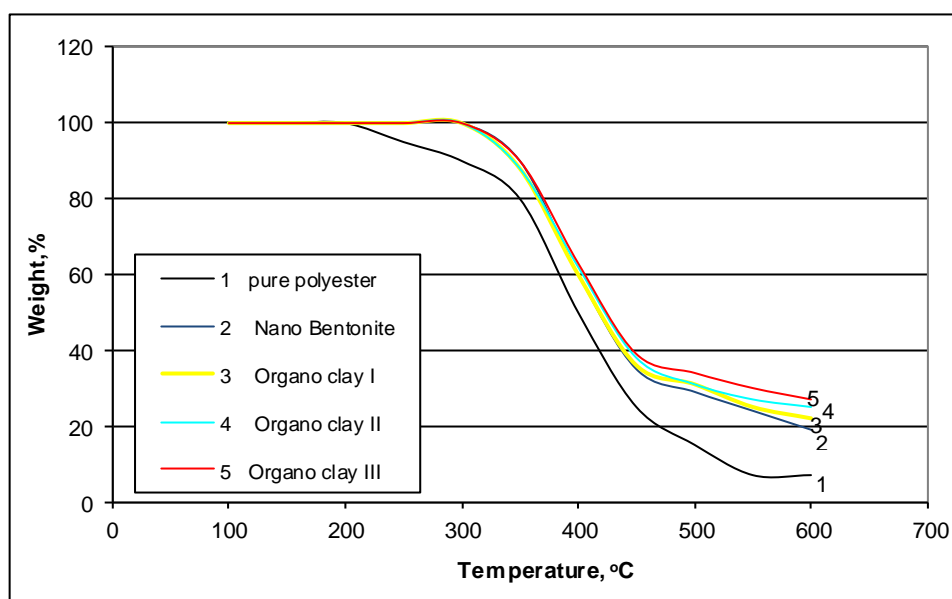


Fig.3 TGA of pure resin and nano composites containing different type of tested material at 5 % weight.

3.2 Morphology Analysis

Morphology analysis of all fabrication nano composite are shown in **Fig.4 (a-g)**. As shown in **Fig.4 a**, the morphology of pure polyester resin is very smooth and amorphous with small wider crack propagation lines. Adding 2% nano bentonite shows distances between the crack propagation lines are much smaller than the pure polyester due to nano bentonite start to mix with polyester but without good dispersion between them as shown in **Fig.4 b**. But by increasing nano bentonite to 5% wt. show a crack propagation lines disappeared but observe some flakes on the surface as show in **Fig.4 c**.

On the other hand nano composite containing 10 wt. % of nano bentonite particles as shown in **Fig.4 d** shows poor adhesion and bonding were observed were not dispersed well and large aggregates on this surface results in easy crack propagation due to increase in nano bentonite particles correspondingly increases viscosity of the modified polyester which act as stress concentrators on the nano composite surface. This causes localized stresses on the surface of the clay leading to failure at mechanical properties at 10wt. % of nano bentonite.

In case of adding of surfactants (I, II and III) to 5%wt. nano bentonite, it showed disappear of all flakes and decrease in interlayer space but observe a large accumulation decrease by increase alkyl chain of surfactant as shown in **Fig.4 (e, f, g)** respectively. So in case of adding the longest alkyl chain (surfactant III) to 5%wt. nano bentonite shows surface with uniform appearance and more smooth structure which give maximum improvement of mechanical and thermal properties as shown in **Fig.4 g**.

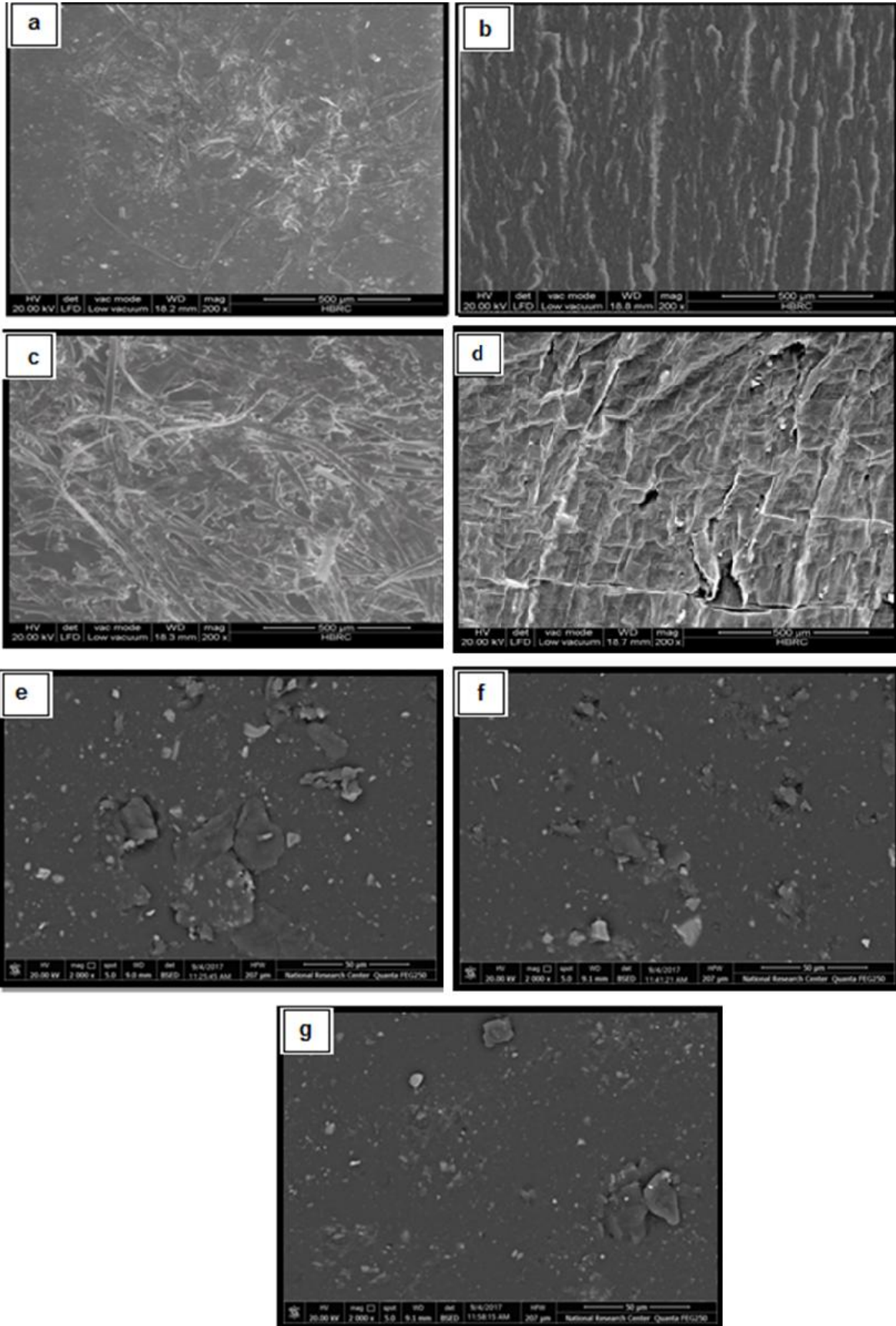


Fig.4 Morphology of nano composites at (a) pure polyester (b) 2% wt. nano bentonite (c) 5% nano bentonite (d) 5%wt. organo clay I (e) 5%wt. organo clay II (f) 5%wt. organo clay III (g) 10% wt. nano bentonite

4. Conclusions

In this paper , thiol surfactants were prepared from the reaction of 2-mercaptopyridine with different alkyl halides with different alkyl chain lengths.

The mechanical, thermal and morphological properties of all fabricated nano composites were studied. From the results obtained the following conclusions were reported:

1. It was observed that, mechanical properties were optimized at 5 wt. % organo clay III content in fabrication nano composite (D). When compared with pure polyester, it gave the following results. Tensile strength showed a 12.5% increase, flexural strength showed a 12.94% increase while the compressive strength showed a 20.42 % increase in strength.
2. The thermal gravimetric analysis (TGA) showed decomposition behavior of adding (nano bentonite, organo clay I , organo clay II and organo clay III) at 5% to polyester which show increasing in decomposition temperature and weight loss constant up to 300 °C when compared with pure polyester resin . It is clear that the best thermal stability of nano composite (D) which formed by adding thiol surfactant (III) to 5% weight nano bentonite.
3. Morphology analysis revealed that excellent adhesion and interfacing between the polyester and organo clay (III) is the main reason for optimum improvement of properties. Finally nano composite (D) improved the mechanical and thermal properties of nano composites which leads to raise the efficiency of polyester resin and can be applied in the manufacture of drinking water pipes.

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المخلص باللغة العربية

تحضير وتقييم بعض المواد ذات النشاط السطحي ومشتقاتها المستخدمة كإضافه لتحسين الخواص الميكانيكية و الحرارية لمواسير مياه الشرب

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1 قسم الكيمياء كلية البنات جامعة عين شمس

2 المركز القومي لبحوث الاسكان والبناء

المخلص

تناولت الدراسة تحضير بعض مركبات عضوية (مواد ذات نشاط سطحي) . وتم تحضير البولي استر / (نانو بنتونيت) مركبة نانوية (أ) ، البولي استر / (عند 5% نانو بنتونيت) /مركب ذات نشاط سطحي (1)مركبة نانوية(ب) ، البولي استر / (عند 5% نانو بنتونيت) /مركب ذات نشاط سطحي (2)مركبة نانوية(ج) ، البولي استر / (عند 5% نانو بنتونيت) /مركب ذات نشاط سطحي (3)مركبة نانوية(د). وقد تم دراسة الخواص الميكانيكية و الحرارية للخلطات السابقة و كذلك استخدام الماسح الألكتروني لدراسة مورفولوجيا المواد التي تم تحضيرها. و أظهرت النتائج أن المواد المركبة التي تم تحضيرها من النانو بنتونيت /بولي استر قد حسنت الخواص عند نسبة 5% من النانو بنتونيت وتقل الخواص بزيادة نسبة النانو بنتونيت. مما سبق يتضح أن استخدام المركبات ذات النشاط السطحي كإضافات الي البولي استر / (نانو بنتونيت عند 5%) تحسن من الخواص الميكانيكية و الحرارية للمركبات النانوية المحضرة ، ولكن مركبة نانوية(د) اعطت اعلي خواص ميكانيكية (قوة) وخواص حراريةوذلك يمكن استخدامها في صناعة مواسير مياه الشرب.