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SiO_2 particles effect on the mechanical properties of the starch/PVA blends

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Abstract

The present work studies the mechanical properties of SiO_2 µPs, and NPs in St/PVA blends. The samples were prepared by casting method as PVA, St/PVA blends at different concentrations (30, 40, 50, and 60 %). DSC and TGA tests were carried out to the samples evolved. The result showed a single glass transition temperature (T_g) for all St /PVA blends that was attributed to the good miscibility of the blends involved. It was found that (T_g) decrease with starch ratio increase. It was seen that (PVA) of (T_g=105 ^{o}C); The glass transition temperature which was decrease with starch ratio that was attributed to glass transition relaxation process due to micro-Brownian motion of the main chain back bond. The endothermic peak at 200 ^{o}C was attributed to melting point of (PVA). Thermal properties of PVA; and St /PVA blends at different concentration (30, 40, 50, and 60 %) were evaluated by thermo gravimetric analysis (TGA). The analyses were carried out from 20 to 600 ^{o}C at 10 ^{o}C)/min heating rate in air oxygen atmosphere. The weight loss stages depended on polymer system. The starch addition causing shifting in the second degradation temperature to the higher temperature; which result in overlapping between the two main degradation steps, these result was attributed to the St/ PVA blend compatibility. The mechanical properties results showed a decrease in ultimate strength with starch ratio increase. The ultimate strength of (PVA) was (47 MPa), whereas the ultimate strength of 60 %St/PVA was (11 MPa) and for 30 %St/PVA was the highest ultimate strength of blends involved (26 MPa). SiO_2µPs (753.7 nm), and NPs (263.1 nm) were added at different concentrations (1.5, 2, and 2.5 %). 1.5% SiO_2µPs, and NPs of the best ultimate strength (69 MPa), (86 MPa) respectively then it was decreased by SiO_2µPs, and NPs increase. Optical microscope of the samples involved was investigated. It was concluded the prepared samples were suggested to be used as packaging materials for agriculture application and its ultimate strength could be controlled by SiO_2µPs, and NPs addition.

Key words
PVA, corn starch, SiO_2, mechanical properties.

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اضافة دقائق اوكسيد السيليكون SiO_2 على الخواص الميكانيكيه لخلائط Starch /PVA

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الخلاصة

يهدف هذا البحث الى دراسة تاثيرNPs و SiO_2µPs على الخواص الميكانيكيه لخلائط St/PVA حضرت النماذج بطريقه الاذابه كنماذج من PVA وخلائط St/PVA بمختلف التراكيز (30, 40, 50, 60 %). اجري فحوصات TGA وDSC على النماذج مدار البحث. اظهرت النتائج وجود درجه انتقال الى الزجاج واحده T_g لجميع خلائط St/PVA ويعزى ذلك الى امتزاجيه جيده للخلائط مدار البحث. وجد تناقص في درجه انتقال ازجاج بزياده تركيز النشا. لوحظ ان درجه انتقال الزجاجPVA, St/PVA تساوي 105 ^{o}C ويعود ذلك الى عمليه الاسترخاء الناجمه عن حركة (micro-Brownian motion) الخاصه بسلسله الرئيسيه للبوليمر. ان قمه الامتصاص الحراري (endothermic) عند 200 ^{o}C تمثل درجه الانصهار ل PVA. حللت الخواص الحراريه PVA و St /PVA لمختلف التراكيز (30, 40, 50, 60 %) بالتحليل الوزني الحراري thermo gravimetric analysis (TGA). اجريه التحليل من 20-600 ^{o}C وبمعدل حراري 10 $^{o}C/min$ في الهواء وجو مؤكسد. رقم المرحله يعتمد على المنظومه البوليمريه. احدثت اضافه النشا درجه من التراكب مابين خطوات التفكك الرئيسي مسببه زحف في درجه حراره التفكك الثانوي نحو درجه حراريه اعلى ويعود ذلك الى توافق النشا مع PVA. اظهرت نتائج الفحوصات الميكانيكيه تناقص في متانة الشد بزيادة نسبه النشا .ان متانه الشد كانت ل PVA مساويه ل (47 MPa) في حين اصبحت متانه الشد ب 60 %St/PVA مساويه (11MPa) وبالنسبه 30 %St/PVA كانت مساويه (26 MPa) وتمثل اعلى متانه شد للخلائط مدار البحث. اضيفت دقائق SiO_2µPs (753.7 nm) و NPs (263.1 nm) للخليط 30 %St/PVA لانه اعطى افضل نتائج شد للخلائط وبتراكيز (1.5, 2, 2.5 %). كانت µPs 1.5 % SiO_2 و NPs تمثل اعلى متانه شد (69 MPa), (86 MPa) على التوالي ثم قلت بزياده تركيز NPs و SiO_2µPs. فسرت النتائج باستخدام المجهر الضوئي. نستنتج بأن نتائج مدار البحث يمكن استخدامها كمواد تعبئه وتغليف للتطبيقات الزراعيه ويمكن السيطره على متانه الشد من خلال اضافه NPs و SiO_2µPs.

Introduction

The drop in the petroleum resources and an exponential growth in the usage of non-biodegradable plastic films cause a great danger to the environment. The composites and polymer blends were development to be biodegradation. Reinforcement of polymer matrices by nano fillers has been studied broadly in the last two decade. SiO_2 particles are white amorphous powder have various linkage of hydroxyl groups on their surface and unsaturated residual bond. Nano and micro SiO2 particulate fillers enhanced the mechanical properties, that attributed to the increase in the contact surface area introduced by the fine particles; and increased the transferred stress. Polyvinyl alcohol (PVA) is a linear artificial polymer formed by partial or full hydrolysis of (polyvinyl acetate) to eliminate the acetate groups. it is a polymer consists of many hydroxyl groups, with very good properties, such as poor permeability, water soluble, good water absorption capability, and used in various industrial and agricultural applications [1-4]. Starch is a semi-crystalline polymer stocked in granules as corn, rice, and potato. It is composed of amylose and amylopectin. The amounts of amylose and amylopectin depend on the source of plant. Corn starch granules generally consist of (70 %) amylopectin and (30 %) amylose [5, 6]. Zainab et al investigated the polymer blend and polymer nano composite that based on (PVA) and studies the Structural properties, barrier properties (The permeability of blends depends on many factors such as the surrounding relative humidity, temperature, film thickness, water and plasticizer contents in addition to the nature of blend components), mechanical, and thermal properties, as well as biodegradation of these material [7]. HanGuo et al found that SiO_2 addition improve the mechanical properties, decreased the degradation as well as in compatibility and Miscibility [8]. Chao et al reported that were increase in tensile strength, amorphous phase and tear strength with SiO_2 addition

[9]. In this work, St/PVA blends were modifying by adding SiO_2 particles to study its effects on the mechanical properties of the blend.

Experimental

The materials used in this research are a white powder of poly vinyl alcohol (PVA), from Barcelona Espana (Didactic). A white powder of corn starch (St) from spain (panreac) with purity (99.9 %), a white powder of SiO_2 µPs (753.7 nm), NPs (263.1 nm) from china, distilled water with purity (100%) to dissolve the PVA, And a beige powder of alpha amylase source (Aspergillus oryzae) from England. (10 g) of purified polymer (PVA) was dissolved in (100 ml) of distilled water in water bath. The solution was mixed by using magnetic stirrer. The stirring process was done for 1 hour, raising the temperature gradually from room temperature (20) ^oC to (90) ^oC and then cooling the solution gradually to room temperature with stirring. The solution was poured on glass plate for curing for 24 hour, after that it can removed by using tong or needle. For preparing St/PVA blend films, the (PVA) was dissolved as previous step and starch was added in different ratio (30, 40, 50, and 60 %) at (90) ^oC, and then cooling the solution to the room temperature for 2 hour with stirring, till get homogeneous solution. The solution was poured on glass plate to curing for 48 hour. For prepared SiO_2/St/PVA films, the solution of (30:70) St/PVA ratio was mixed with silicon dioxide (SiO_2) powder at (90) ^oC and then cooling to room temperature for 2 hour. The silicon dioxide was added in two particle size micro particle (SiO_2 µPs), and nano particle (SiO_2 NPs) in different ratios (1.5, 2, and 2.5), to study the effect of particle size on the blend. Differential scanning calorimetry (DSC) was performed using a STA PT 1000 from Linseis Germany. The weights were within (16-21) mg cut in circle plates, packed in platinum pans and sited in the DSC cell. The heating rate was (10) ^oC/min, the resulting curve represents the relationship between temperature (^oC) in x-axis and HDSC (heat flow) (µV) in y-axis. Thermo gravimetric analysis (TGA) was performed using a STA PT 1000 from Linseis Germany. The samples were carried out from 65 to 600 ^oC at a heating rate of (10) ^oC/min, in air. The weight of the sample was between (16- 21) mg. The given data represents the relationship between temperature (^oC) in x-axis and Rel-mass change (%) in y-axis. Tension test is used to determine the mechanical properties of the samples. It is broadly used to get main design data on the material strength. It is a suitable test to measure the material requirement. The main parameters that establish the stress-strain curves are the yield strength, elastic modulus, ultimate strength, and F_{max}. The samples were characterize by tensile machine of (LARYEE-50 KN, China), to determine the ultimate tensile strength (UTS), elongation (%), and young modulus (E). The samples were cut according to standard (ASTM D-638). The speed of cross head was (5 mm/min). The test was done at room temperature. Optical microscopes are widely used for sample examination because of their low price, simple sample preparation and ease of use. It is used to study cracks, air bubbles and deformation in samples. The morphology of PVA, St/PVA blend, and µPs, and NPs SiO_2/St/PVA films were studied using Optical microscope of Carl Zeiss Company, with magnification X100 (100 times magnified).

Results and discussion

1.Differential Scanning Calorimetry (DSC)

Figs. 1 to 6 show the biodegradation reaction occurred during glass transition temperature (T_g) and melting temperature (T_m)[10]. It was found decrease in (T_g) with starch ratio increase, whereas increase in melting point (T_m). The result proved one glass transition temperature (T_g) for all St/PVA blends that was attributed to the good miscibility of the blends involved. The miscibility was due to the hydrogen bonds between the (PVA) and (Starch).this miscibility was associated with hydrogen bonds between the hydroxyl groups (OH) of (PVA) and (Starch). The (T_g) and (T_m) values were listed in Table 1. It was seen that (T_g) and (T_m) values were plasticizer contents dependent. It was found that decrease (T_g) with starch ratio increase which is in a good agreement with (Shafik, and Esraa) [10-13]. The role of starch was (T_g) decrease that was in a good agreement with [Fahmide] [14]. Poly vinyl alcohol is composed of crystalline and amorphous phases, the result in quite a complex behavior when the polymer is heated. From the previous Fig.2., it was found that (PVA) glass transition temperature (T_g =105 ^oC); which attributed to the glass transition (T_g) relaxation process due to micro-Brownian motion of the main chain back bond [13]. The endothermic peak at 200 ^oC was attributed to melting point of (PVA) which is in a good agreement with (Esraa and Elisabita) [13-15].

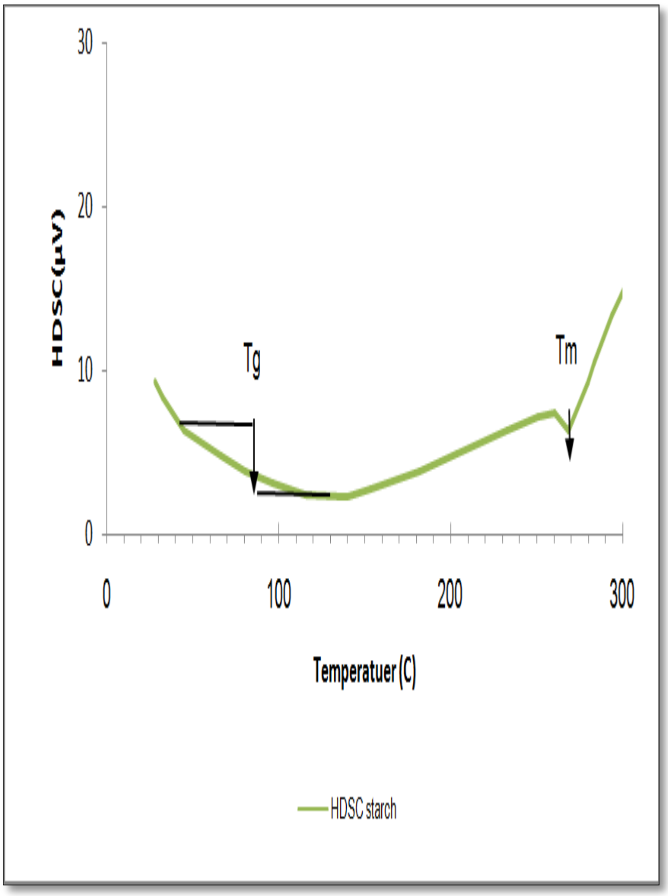


Fig.1: DSC of pure starch.

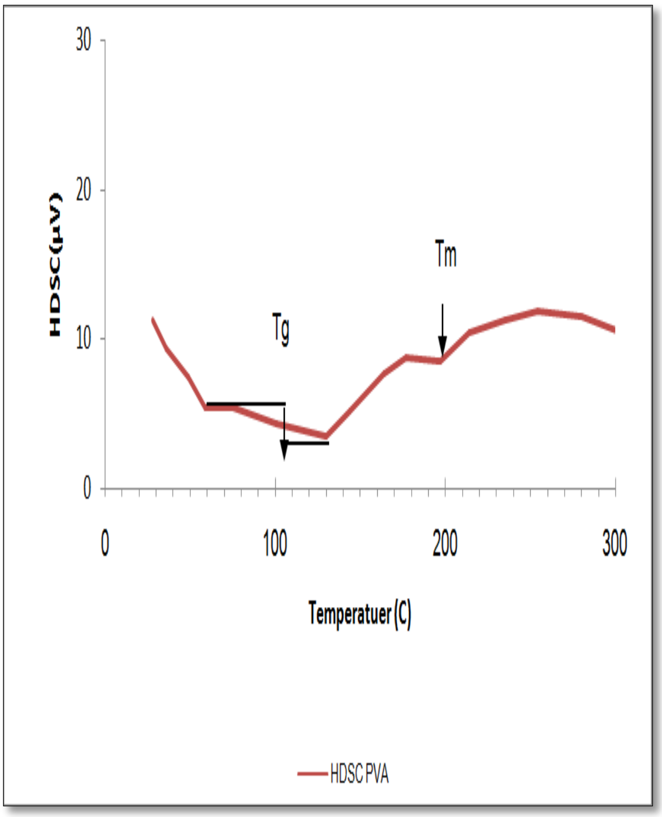


Fig.2: DSC of pure PVA.

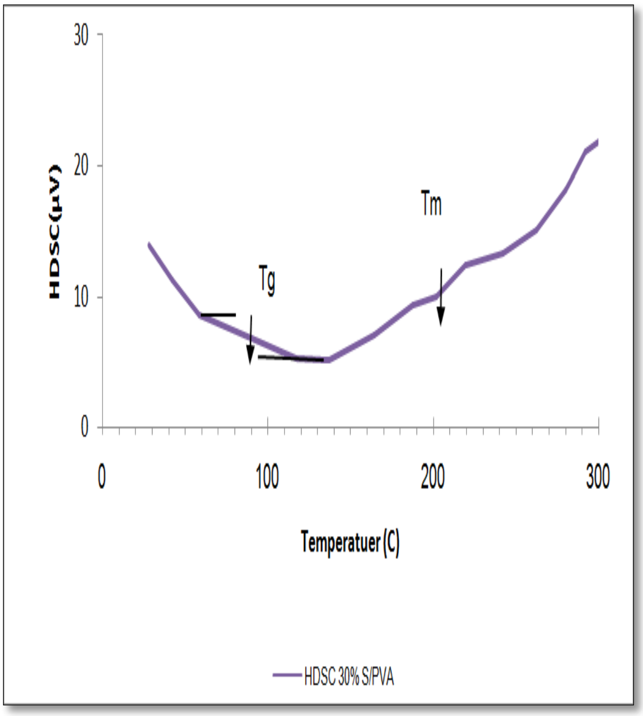


Fig. 3: DSC of 30 %St/PVA.

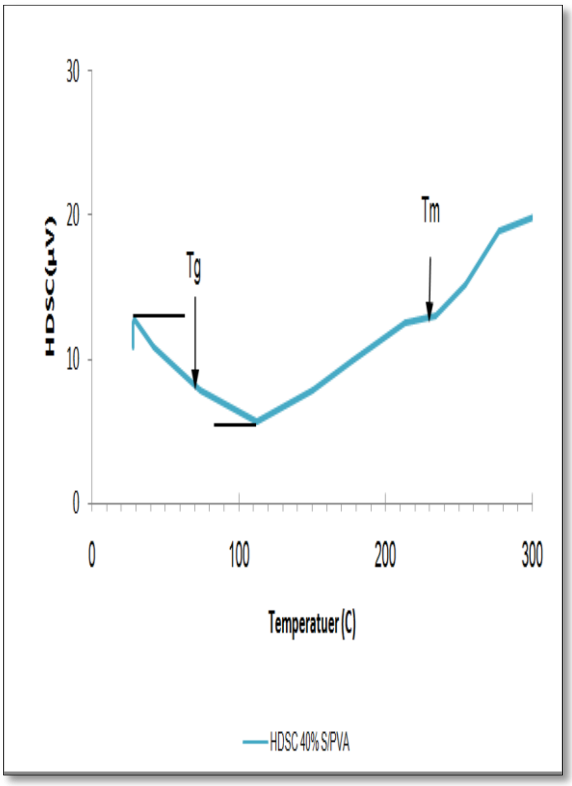


Fig.4: DSC of 40 %St/PVA.

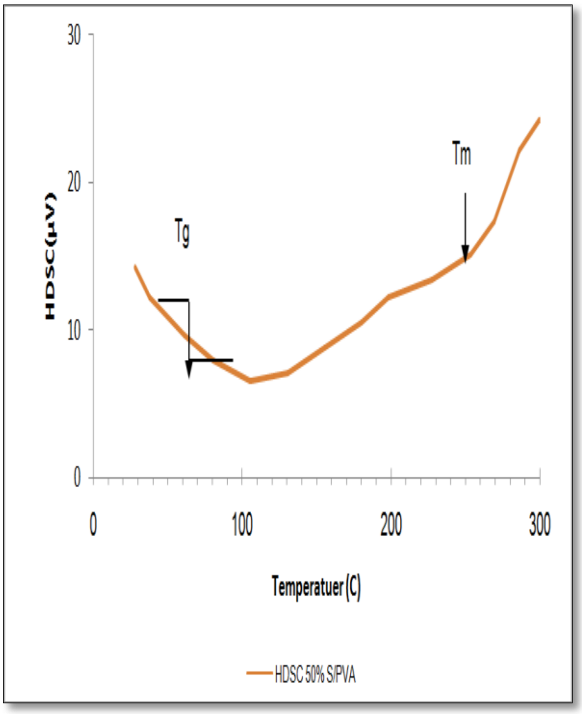


Fig. 5: DSC of 50 %St/PVA.

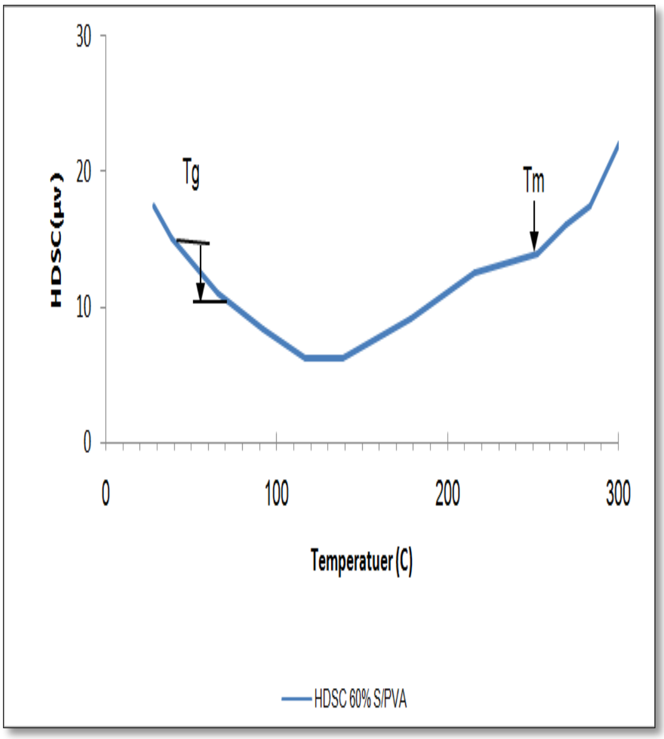


Fig.6: DSC of 60 %St/PVA.

Table 1: Shows T_g and Tm values.

Polymer system	T_g °C	T_m °C
Starch	85	270
PVA	105	200
30% St/PVA	90	205
40% St/PVA	70	230
50% St/PVA	65	250
60% St/PVA	55	250

2.Thermo Gravimetric Analyses (TGA)

Thermal properties of PVA; and St /PVA blends at different concentration (30, 40, 50, and 60%) were evaluated by thermo gravimetric analysis (TGA). The analyses were carried out form 20 to 600 °C at 10 °C/min heating rate in air oxygen atmosphere. Fig. 7 to 12 summarizes the (TGA) results of the samples involved. The number of stage is polymer system dependent. Table 2 shows the weight loss stages of (PVA) occurred primarily in three stages. The first one was within range of (210-373.02 °C); the second one was within (373.02-460 °C); and the third was (460-592.3); which were corresponded in weight loss of (40.68, 33.7279, and 15.917 %), respectively. The first loss was due to the evaporation of residual moisture; the second loss, which corresponded to the side chain decomposition of PVA, and the third correspond to the main chain of PVA [12]. The result showed that the onset temperature of degradation reaction of PVA was significantly reduced by starch addition [15]. The degradation of blend started at lower temperatures was attributed to the degradation of interface of St/PVA. The starch addition caused some degree of overlapping between the two main degradation steps, causing a shift of the second degradation temperature to the higher temperature; that was attributed to the compatibility of Starch; with PVA; where in thermally resistant cyclic hemiacetal in starch imparts thermal stability to the St/PVA blend [16]. When starch was added of different concentration, the stability of blend lowered down compared to the pure PVA. It was concluded that starch addition to the (PVA) reduced the thermal stability of PVA [17]. For (30 % St/PVA); the disappearance of the second and third stages was due to the decrease in the volatilization of starch [18].

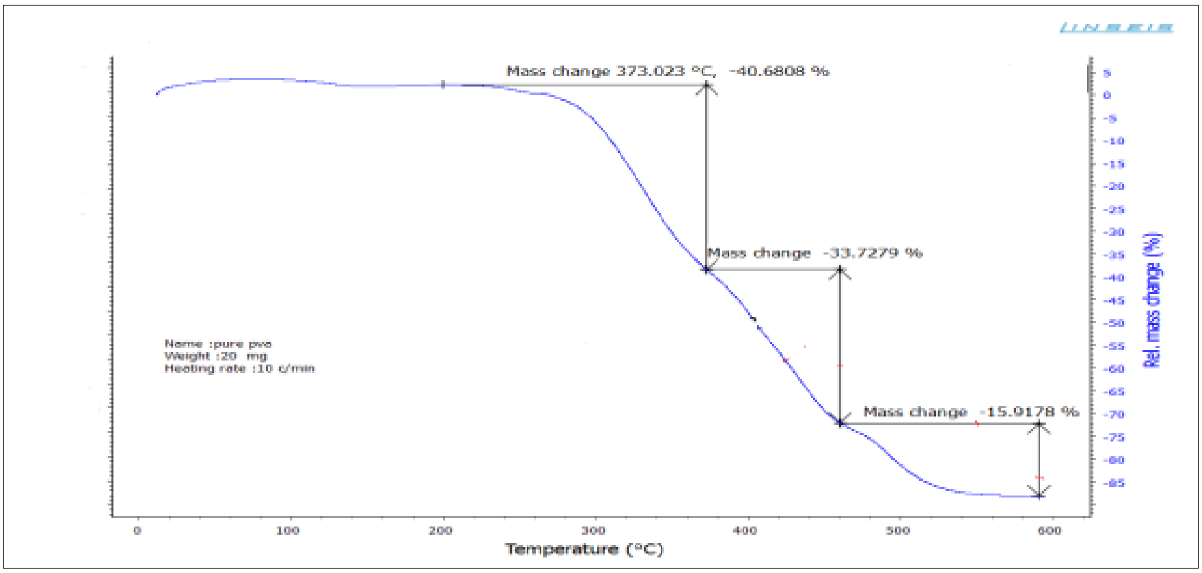


Fig. 7: TGA pure PVA.

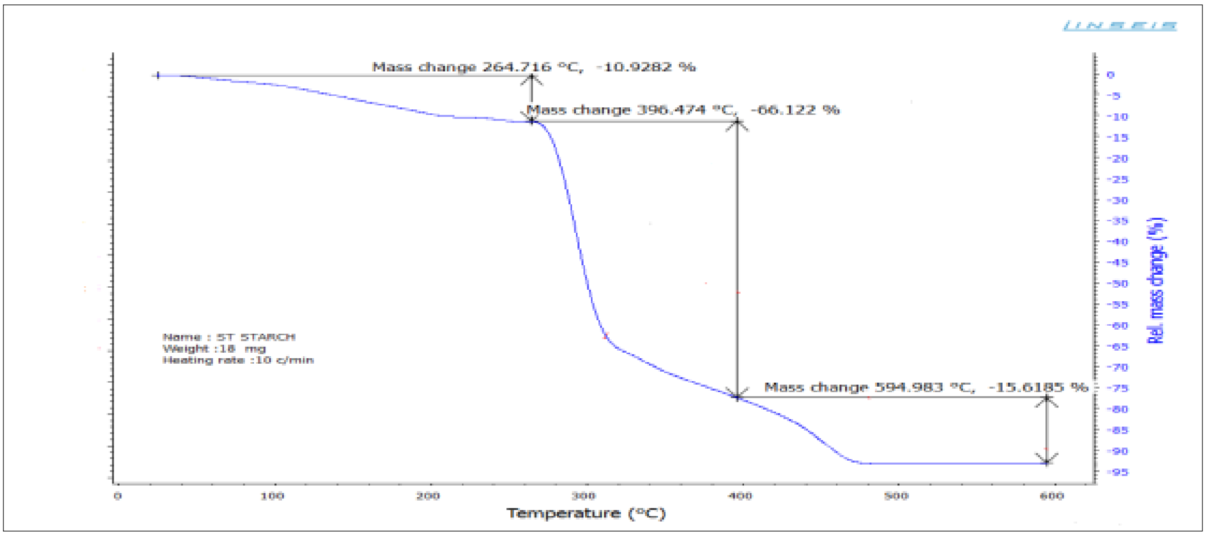


Fig. 8: TGA pure starch.

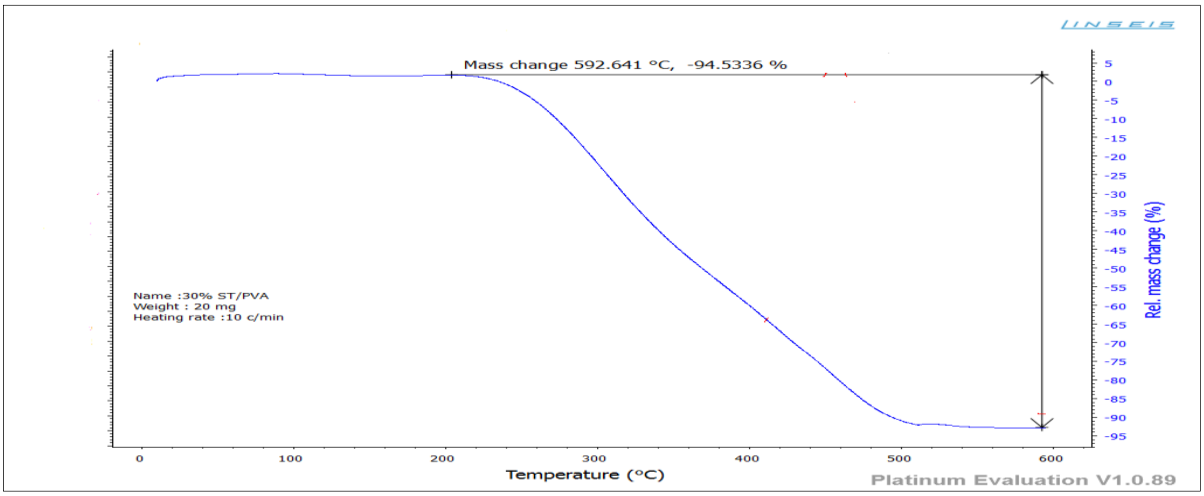


Fig. 9: TGA 30 %St/PVA.

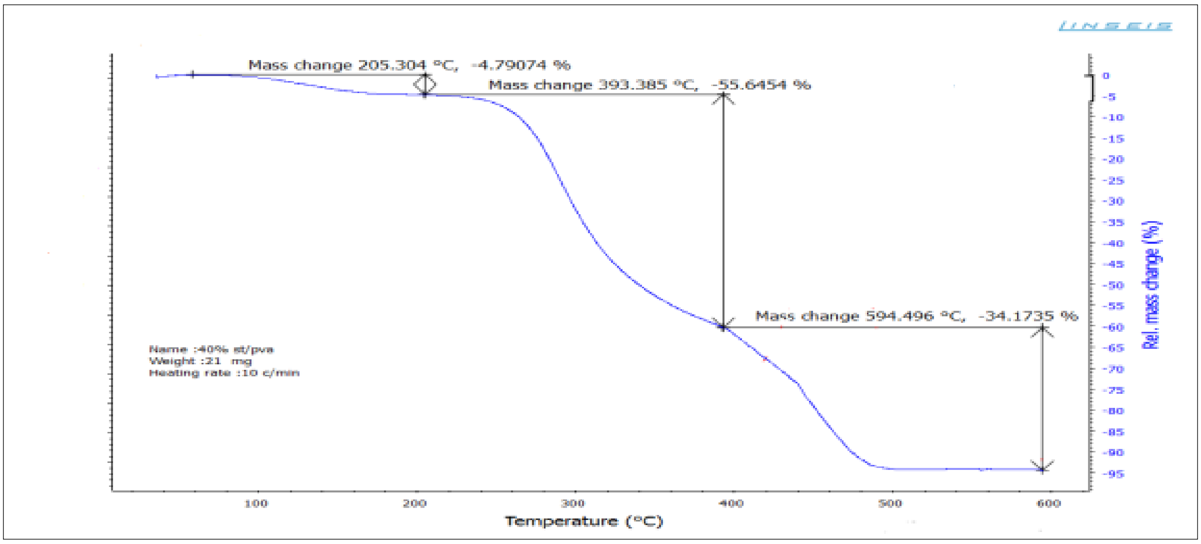


Fig. 10: TGA 40 %St/PVA.

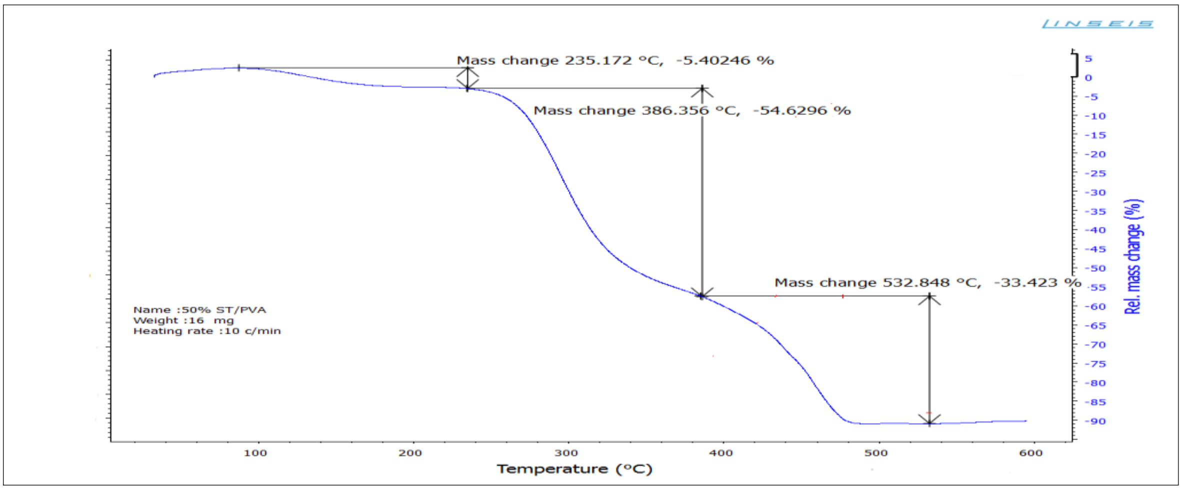


Fig. 11: TGA 50 %St/PVA.

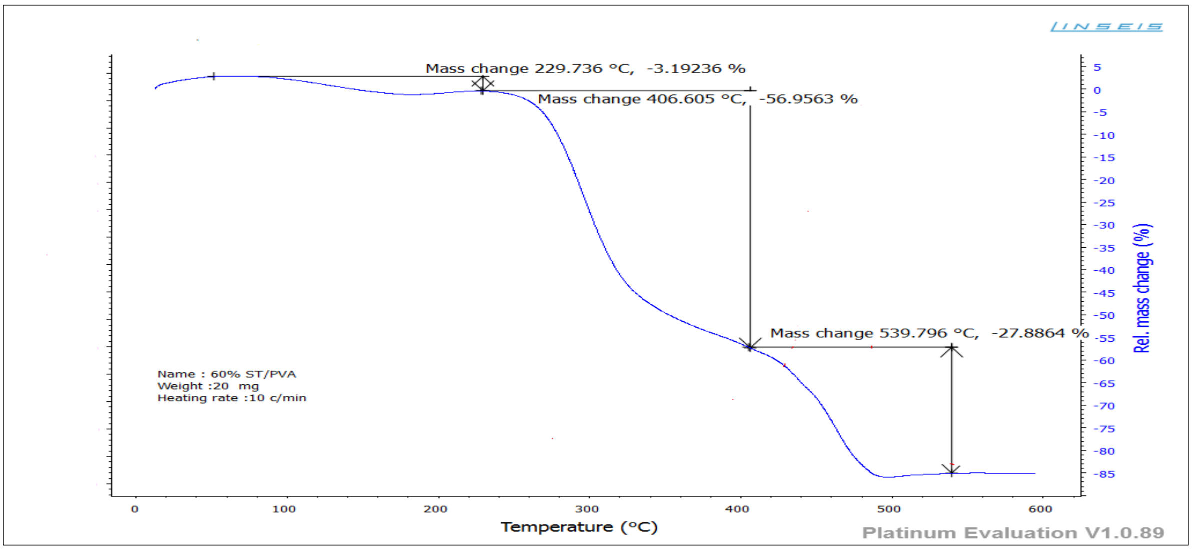


Fig. 12: TGA 60 %St/PVA.

Table 2: Thermal decomposition data of the St/PVA blend film.

Polymer system	*First stage*		*Second stage*		*Third stage*		*IDT °C*	*PDT $_{max}$*
	T	*Weight loss%*	*T*	*Weight loss%*	*T*	*Weight loss%*		
PVA	210- 373.023	40.6808	373.02-460	33.72790	460-592.3	15.917	373.02	460
Starch	20-264.716	10.9282	264.716-396.474	66.122	396.474-594.983	15.6185	264.716	495
30%St/PVA	205-592.641	94.5336	/	/	/	/	205	592.64
40%St/PVA	60-205.304	4.79074	205.304-393.385	55.6454	393.385-594.496	34.1735	205.304	395
50%St/PVA	95-235.172	5.40246	235.172-386.356	54.6296	386.356-532.848	33.423	23.517	390
60%St/PVA	62-229.736	3.19236	229.736-406.605	56.9563	406.605-539.796	27.8864	229.736	406.65

3. Mechanical properties

Fig. 13 shows stress- strain curve for (PVA) and St/PVA blended at different concentrations (30, 40, 50, and 60 %). The result showed a decrease in ultimate strength with starch ratio increase Fig. 14 which is in a good agreement with Fahmide and Esraa [13, 19]. These results were attributed to decrease in hydroxyl group by starch addition hydroxyl group give good mechanical properties to polymer system. Starch also possess hydroxyl group (OH), which were reduced during (actylation process). The hydroxyl group decreased caused ultimate strength decreased; that was a good agreement with Fahmide and Qingjie [14, 20]. The starch addition to (PVA) caused a fall in tensile strength; that was due to the decrease in hard segment contents and hydrogen bonding density in the films; this was in a good agreement with

[12,16]. Fig. 15 and 16 show the stress-strain curve of 30 %St/PVA after SiO_2 µPs, and NPs addition at different concentration (1.5, 2, and 2.5 %). The result proved that the tensile strength of the (PVA) was (47MPa) which was higher than St/PVA film at different ratio. Whereas there was significant increase in tensile strength by SiO_2 (µPs, and NPs); which is attribute to formation of intermolecular bonds between (SiO_2-PVA) and (SiO_2-Starch), 1.5 % SiO_2 (µPs, and NPs) of the higher tensile strength value. It was (75 MPa) for 1.5% SiO_2 (µPs) addition and (86 MPa) for 1.5 % SiO_2 (NPs) addition Fig.15 and 16. The tensile strength decrease due to saturations of intermolecular bonding at high concentration of SiO_2 µPs, and NPs Fig. 17a and 17b; which is in a good agreement with [21]. It was found that tensile strength at nano SiO_2 particles addition was higher than that of micro SiO_2 particles addition Fig. 18; that was attribute to the fact of large particles and dispersion- strengthened composites are the two sub classification of particle-reinforced composite. The distinction between these is boned–up on reinforcement or, strengthening mechanism. The term "large" or "micro" is used to indicate the particle-matrix interaction cannot be treated on the atomic or molecular level; rather, continuum mechanics is used. For most of these composites, the particulate phase is harder and stiffer than the matrix. These reinforcing particles tend to retain movement of matrix Phase in the vicinity of each particle. In essence, the matrix transfers some of the applied stress to the particles which bear a fraction of the load. The degree of reinforcement or improvement of mechanical behavior depends on the strong bonding at matrix-particle interface. For dispersion-strengthened composite, particle diameters within range of (10-100 nm). The matrix interaction strengthened on the atomic or molecular level. The mechanism of strengthened is similar to that for precipitation hardening. Whereas the matrix bears the major portion of an applied load, the small dispersion particles hinder or impede the motion of dislocation. The, plastic deformation is restricted such that yield and tensile strength, improved [21]. Figs. 19-23 show the elongation of samples involved, it was seen decrease in elongation with starch, and SiO_2 µPs, and NPs addition: whereas significant increase in young modules Fig. 24-26. This may lead to efficient load transfer between the matrix and filler, causing enhancement in mechanical properties [12].

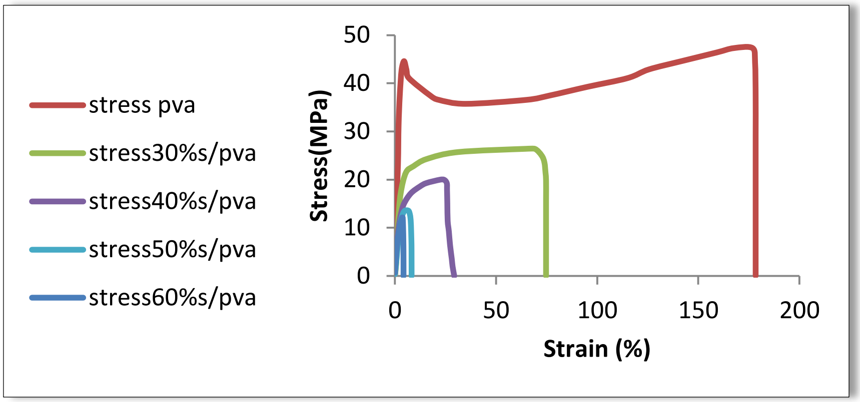


Fig. 13: Stress-strain curve of St/PVA blend film.

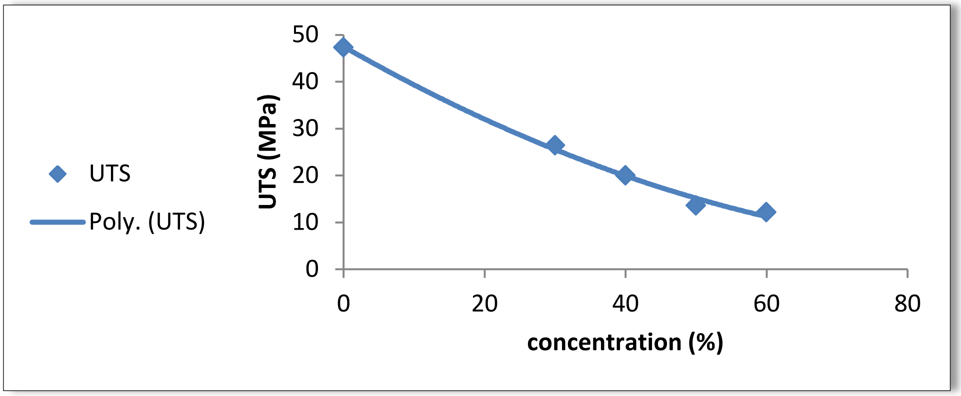


Fig. 14: Ultimate strength of St/PVA blend film.

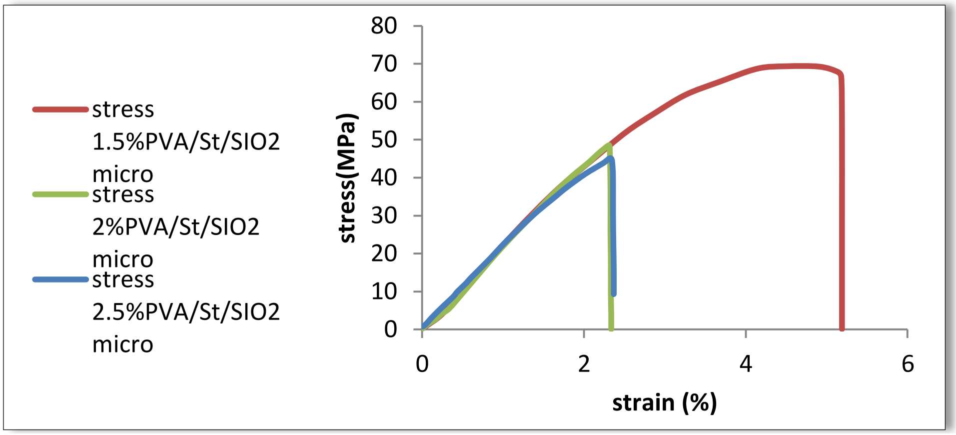


Fig. 15: Stress-strain curve of micro SiO_2/St/PVA blend film.

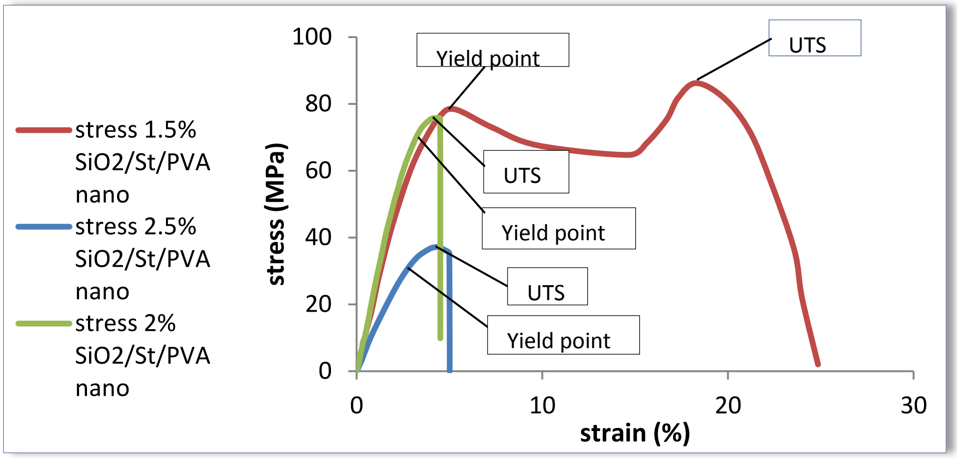


Fig. 16: Stress-strain curve of nano SiO_2/St/PVA blend film.

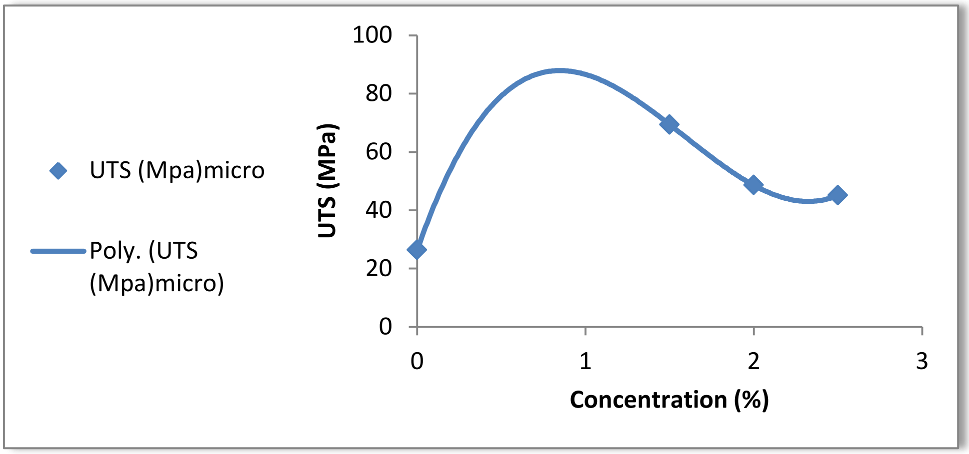


Fig. 17a: Ultimate strength of micro SiO_2 / St/PVA blend film.

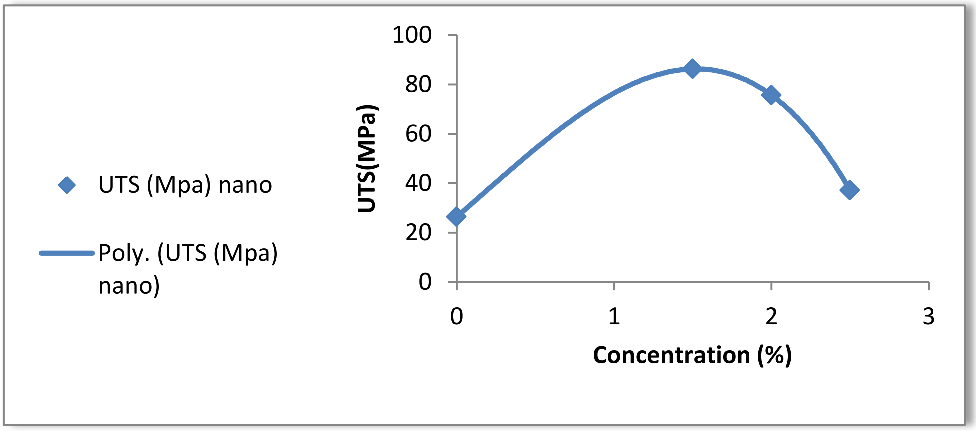


Fig. 17b: Ultimate strength of nano SiO_2 /St/PVA blend film.

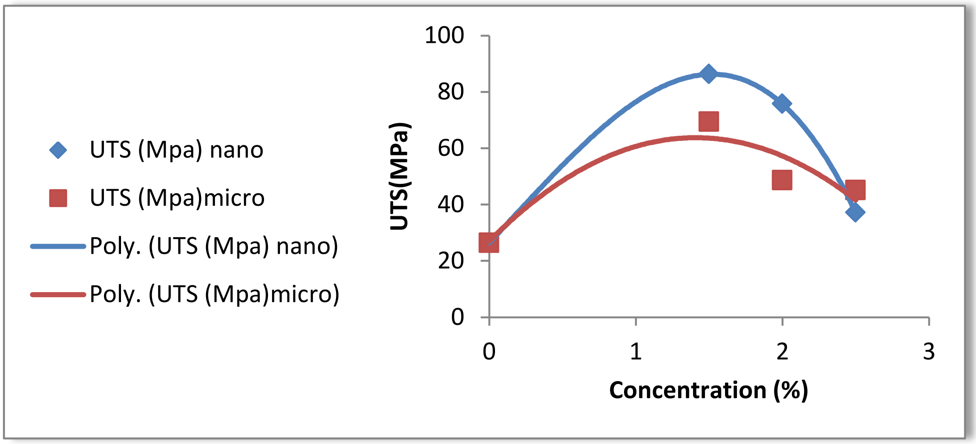


Fig. 18: Ultimate tensile strength of SiO_2 /St/PVA film.

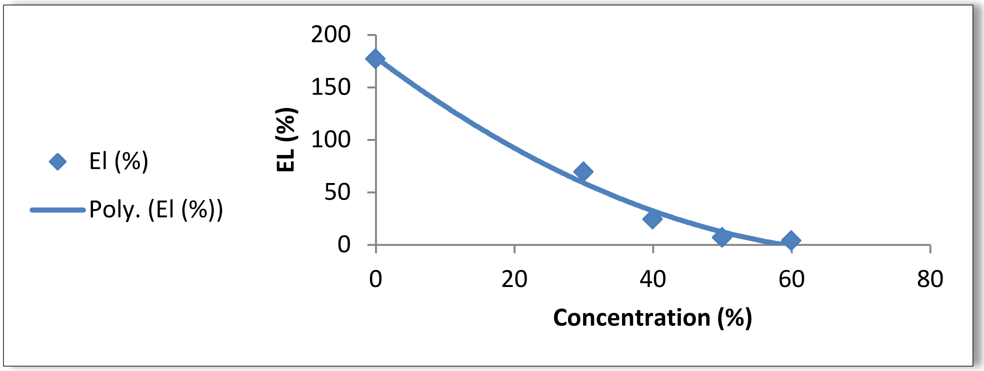


Fig. 19: Elongation of St/PVA blend film.

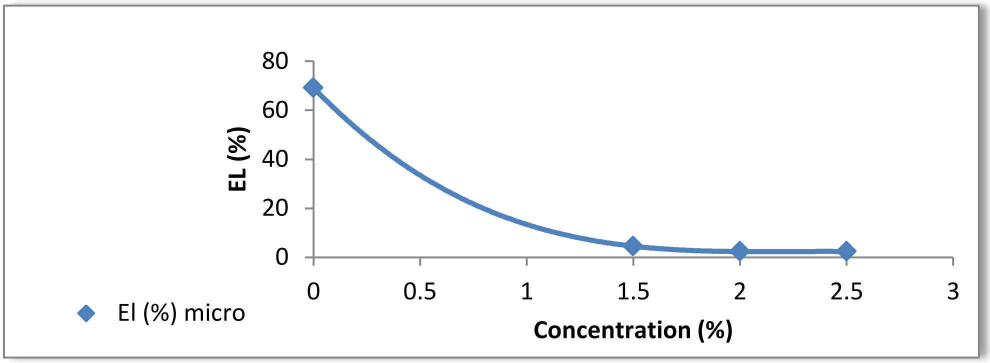


Fig 20: Elongation of micro SiO_2 /St/PVA blend film.

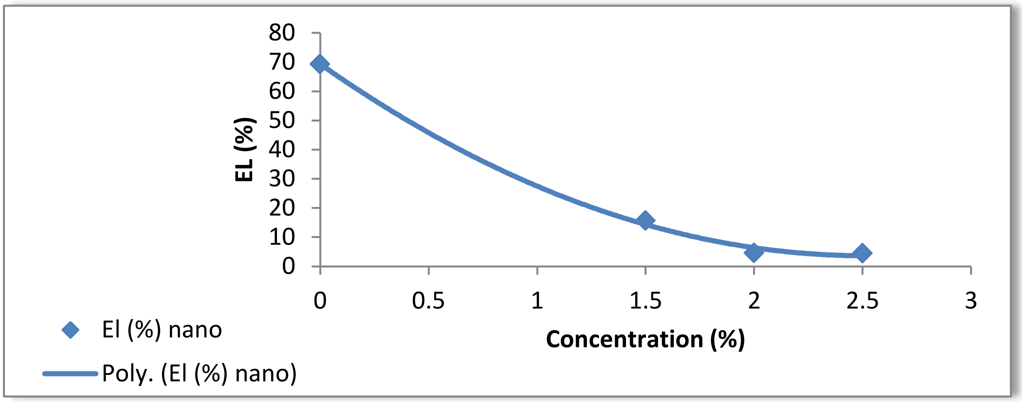


Fig. 21: Elongation of nano SiO_2 /St/PVA blend film.

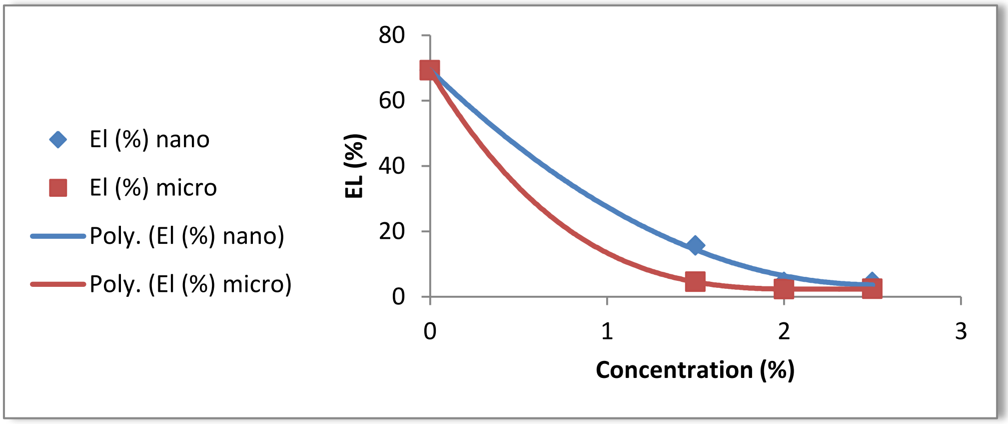


Fig. 22: Elongation of SiO_2 /St/PVA film.

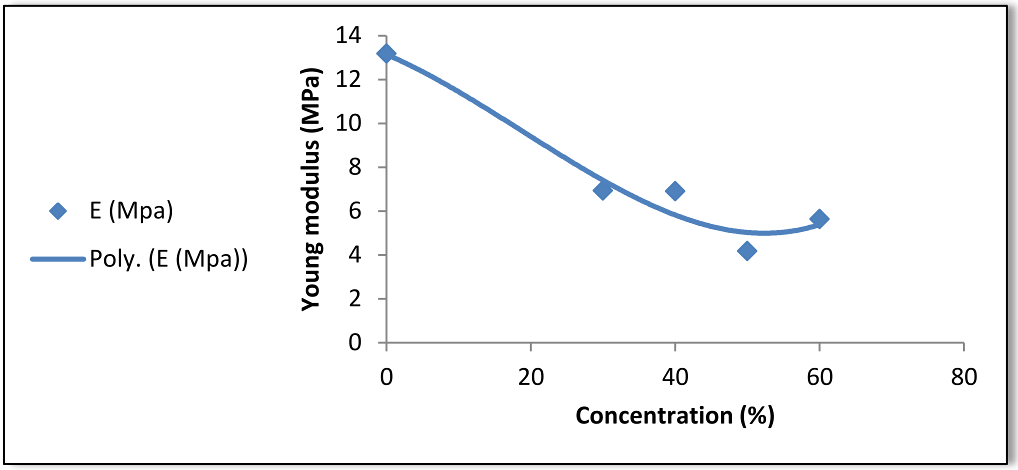


Fig. 23: Young modulus of St/PVA blend film.

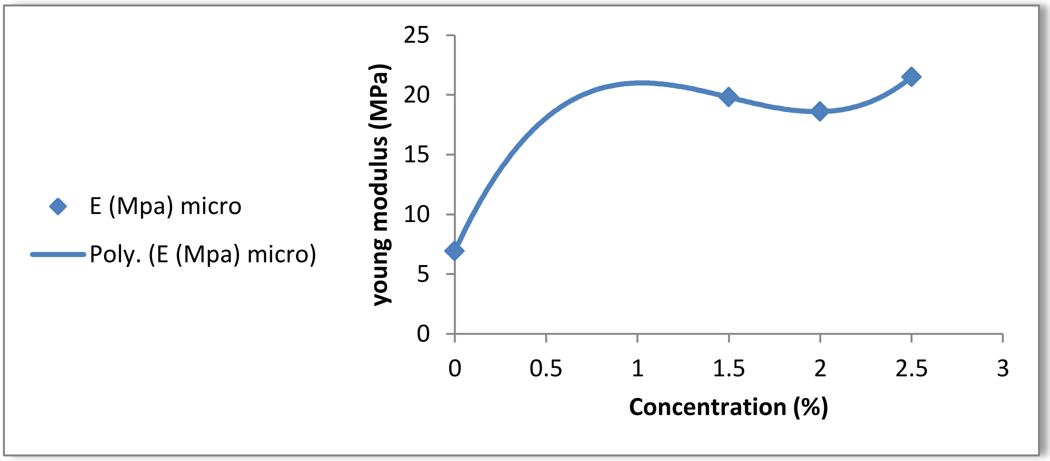


Fig. 24: Young modulus of micro SiO_2 /St/PVA blend film.

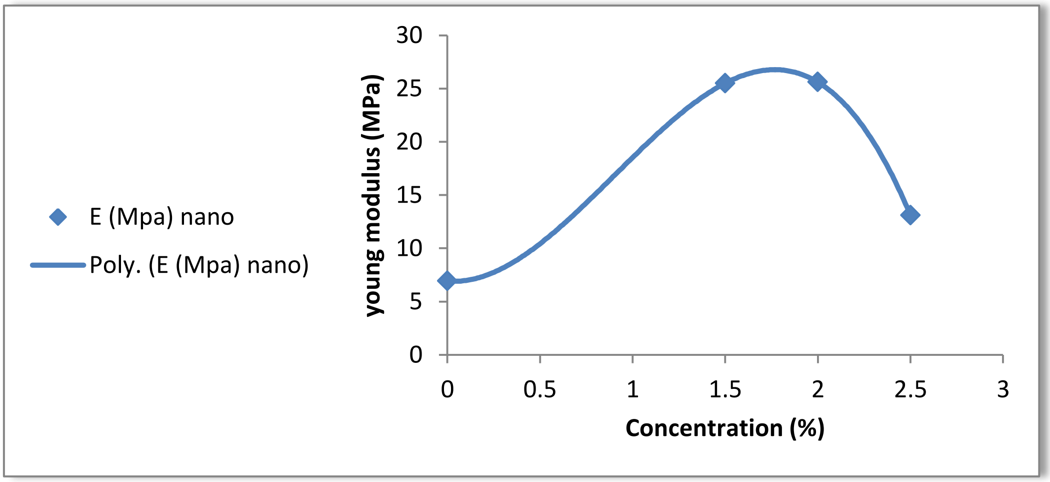


Fig. 25: Young modulus of nano SiO_2 /St/PVA blend film.

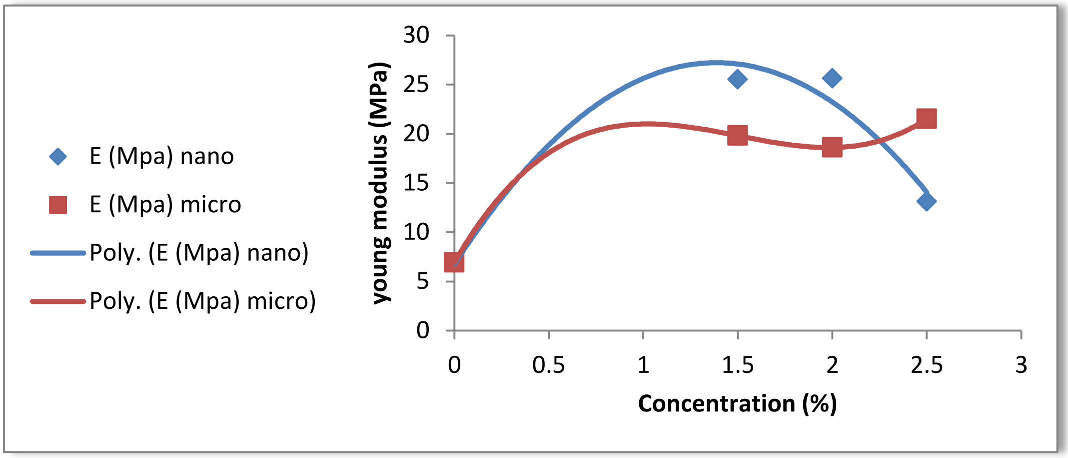
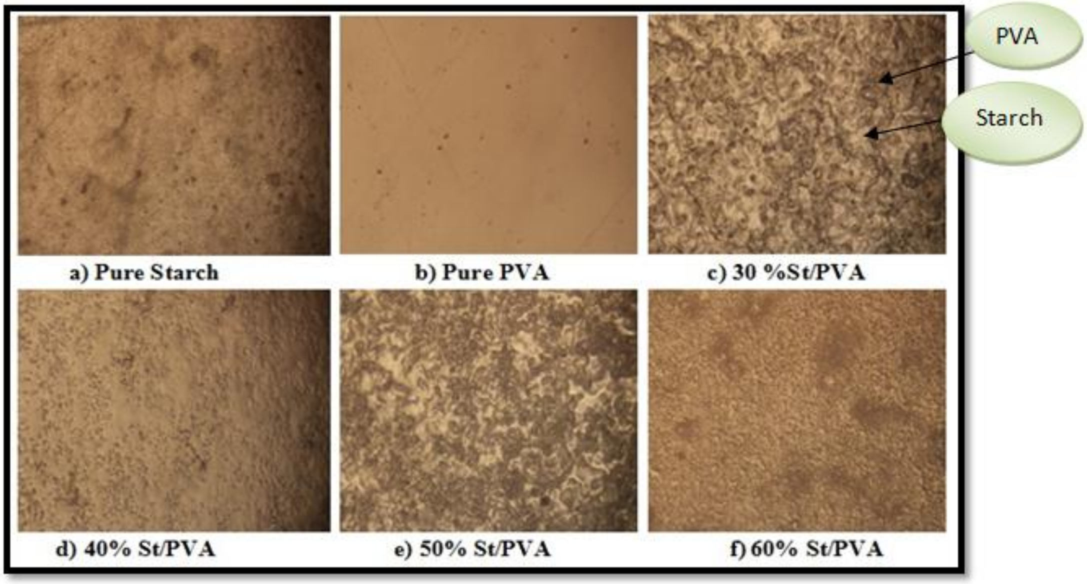


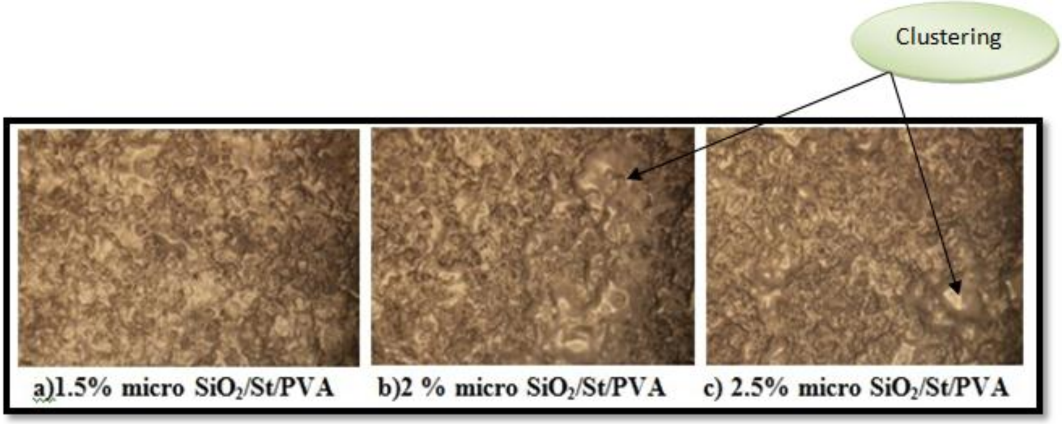
Fig. 26: Young modulus of SiO_2 /St/PVA film.

These results was enhanced with optical micrographs, Figs. 27 to 29 show the optical micrographs of (PVA), (Starch), and St/PVA blends at different concentration (30-60%) before and after SiO_2 µPs, and NPs addition at different concentration (1.5, 2, and 2.5%). the results proved that the blend compatibility increased with starch ratio and SiO_2 NPa concentration, but decreased with SiO_2 µPs addition. That was in a good agreement with [23]. By starch addition; the dispersed phase (starch) would be miscible in (PVA); as a results of that the blends would be miscible blends and of homogenous morphology; it was attributed to the (PVA) characteristic (hydrophilic); that property caused hydrogen linking increase and result in an adhesion strength between the starch chain. This results in a good agreement with Azahari [24]. It was seen the porous increased with (PVA) ratio increased that was attributed to the blend miscibility that was in a good agreement with Esraa [13]. As shown in Fig.29 for NPs SiO_2/St/PVA at different concentration; that the (SiO_2) NPs films were smooth and compact that suggested the miscibility and compatibility of samples involved increased. Because the surface of NPs SiO_2 had plenty of unsaturated residual bonds, and different hydroxyl group bonding stats; it was ease to form a strong hydrogen bond with starch and (PVA). The Strong chemical bond (C-O-Si) was also formed in NPs SiO_2 /St/PVA hybrid materials, such that the strong interfacial binding force took place in NPs SiO_2 and St/PVA, and the films surface showed compact and smooth ultra structure [8].



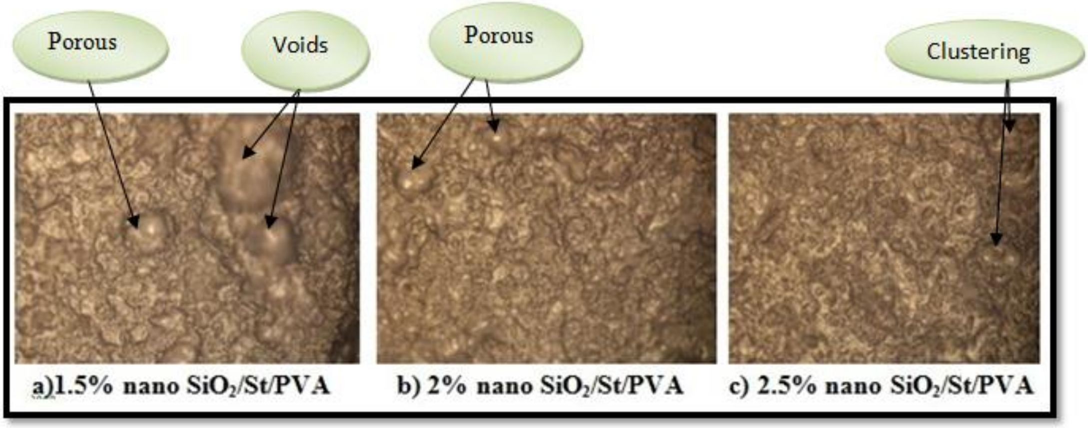
a) Pure Starch b) Pure PVA c) 30 %St/PVA
d) 40% St/PVA e) 50% St/PVA f) 60% St/PVA

Fig. 27: The optical micrographs of (PVA), (starch), and (St/PVA) blend films.



a)1.5% micro SiO_2/St/PVA b)2 % micro SiO_2/St/PVA c) 2.5% micro SiO_2/St/PVA

Fig. 28: The optical micrographs of (micro SiO_2/St/PVA) films.



a)1.5% nano SiO_2/St/PVA b) 2% nano SiO_2/St/PVA c) 2.5% nano SiO_2/St/PVA

Fig. 29: The optical micrographs of (nano SiO_2/St/PVA) films.

Conclusion

In this work, the results showed a single glass transition temperature (T_g) for all St /PVA blends that was attributed to the good miscibility of the blends involved. The ultimate strength of (PVA) was (47MPa), whereas the ultimate strength of

60%St/PVA was (11 MPa) and for 30%St/PVA was the highest ultimate strength of blends involved (26 MPa).1.5% SiO_2µPs, and NPs of the best ultimate strength (69 MPa), (86 MPa) respectively then it was decreased by SiO_2µPs, and NPs increase. Optical microscope result proved that porous increase with (PVA) ratio increase that was attributed to the blend miscibility. For NPs SiO_2/St/PVA at different concentration: the (SiO_2) NPs films were smooth and compact that suggested the miscibility and compatibility of samples involved increased. It was concluded that the samples involved could be used as packaging materials for agriculture application and its degradation could be controlled by SiO_2µPs, and NPs addition.

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