

Optimization Conditions For Preparation of Polyvinyl Alcohol-Alumina Gel Composite And Its Properties

Raghad.U.Abass*

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Abstract

A preparation of poly (vinyl alcohol) (PVA)/alumina (Al_2O_3) gel composite was investigated.

This type of composite gel was carried out with two stages firstly the hydrolysis of polyvinyl alcohol in a hot water at 200°C for 2 hours and different mixing ratio of (PVA/ H_2O) solution at (10,20,30,40,50) wt% has been investigated to check its operating conditions .Secondly different ratios of alumina refractory were used (5,10,15,20,25) wt% of (Al_2O_3 /PVA) at 350°C for 4 hours to give a completes composite gel of (PVA/ Al_2O_3) in a polymerization stage.

The final composite gel of (Al_2O_3 /PVA) is molded in a different shapes and size due to suitable characteristics tests in order to check chemical, thermal, and mechanical properties afterward curing these specimens at 120°C for 2 hours to complete the compatibility of composite gel.

The results tests proved that: All final composite gel specimen is more efficient than other base polyvinyl alcohol (PVA) of high mechanical and thermal properties than base one with less internal stresses, also the resistance for chemical solutions in (100% H_2O , 10% H_2SO_4) is increased at sever conditions at 50°C for 5 days especially for optimum mixing ratio specimen No.3 with preference for optimum sample No.3 of (30%PVA Sol./15% Al_2O_3) g/g.

Keywords: Optimization conditions , preparation, polyvinyl alcohol, alumina gel composite, properties

الظروف المثلى لتحضير المترابك الجلاتيني (بولي فاينيل الكحول – الومينا) و خواصه

الخلاصة

تم تحضير ودراسة المترابك الجلاتيني (Al_2O_3 /PVA) (بولي فنيل الكحول/الالومينا) . تم تحضير هذا المترابك الجلاتيني (Al_2O_3 /PVA) بمرحلتين رئيسيتين المرحلة الأولى هي مرحلة التحلل المائي لبوليمر بولي فاينيل الكحول في محلول مائي عند درجة حرارة 200°C لمدة ساعتان وباستخدام نسب وزنيه مختلفة لهذا البوليمر (PVA/ H_2O) (10,20,30,40,50) % لمعرفة الظروف العملية المثلى. إما المرحلة الثانية فهي لتحضير المترابك الجلاتيني عن طريق استخدام بلمرة المحلول المائي لفاينيل الكحول بإضافة نسب وزنيه مختلفة لمادة الالومينا Al_2O_3 السيراميكية وبنسب خلط (5,10,15,20,25) wt % عند درجة الحرارة 350°C ولمدة أربع ساعات لإعطاء الشكل النهائي لهذا المترابك الجلاتيني (Al_2O_3 /PVA).

وفيما بعد يجري صب المترابك الجلاتيني النهائي بأشكال وحجوم مختلفة ليناسب الفحوصات التطبيقية للمترابك وهي (الخصائص الكيماوية , الحرارية , والميكانيكية) فيما بعد انضاج المركب النهائي عند درجة حرارة 120°C لمدة ساعتين لإتمام التجانس النهائي للمترابك الجلاتيني. إن نتائج الفحوصات أوضحت الكفاءة العالية للمترابك الجلاتيني ذو الأساس البوليمري (Al_2O_3 /PVA) لكل من الخصائص الميكانيكية والحرارية عن البوليمر القياسي PVA وباقل اجهادات.

إضافة إلى مقاومتها الكيماوية داخل المحاليل الكيماوية وهي (100%H₂O, 10%H₂SO₄) والتي أنجزت عند درجة حرارة 50°C ولمدة خمسة أيام أثبتت النتائج لجميع الخلطات وبالأخص الخلطة المثالية النموذج (3) (30% PVA Sol./15% Al₂O₃) والتي أعطت أفضل الخصائص التطبيقية.

Introduction

Membrane pervaporation for the separation of liquid chemical mixtures is a research area of growing interest and importance. [1] Currently, most commercial pervaporation processes involve dehydration of alcohols and other organic solvents, or recovery of organic solvents from dilute aqueous streams. In recent years the potential use of pervaporation technology for the separation of organic mixtures (especially isotropic or close boiling mixtures) has attracted significant attention in the research community. To date, the use of commercially available polymeric membranes in the above application has not attained widespread industrial acceptance owing, in part, to degradation of membrane performance in organic mixtures which cause swelling and loss of membrane integrity [2, 3].

In order to improve membrane stability, the common approach proposed in the literature has been to utilize cross linked [4, 5, 6] and/or blended polymeric membrane materials [5, 6, 7, 8, 9] that are optimized for specific chemical mixtures and operating conditions. An alternate approach is the adaptation of ceramic materials as a stable support structure for the polymeric membrane, thereby providing sufficient chemical and thermal stability to withstand anhydrous solvents and higher temperatures. The emergence of robust inorganic membranes of controlled pore size

[10] has resulted in the creation of ceramic-supported polymer (CSP) membranes in which a polymer layer provides the separation performance of the membrane, while the inorganic support provides the desired mechanical integrity a different approach was taken by Jou et al. [11] who reported on a class of organic-selective pervaporation membranes prepared by a two-step surface free-radical graft polymerization process. Pervaporation membranes consisting of an active layer of PVAc chains terminally and covalently anchored onto the surface of a 500 Å pore size silica support membrane performed with separation factors of up to 106 and 118 for aqueous solutions of trichloroethylene (TCE) and chloroform (500-700 ppm) respectively, with permeation fluxes of up to 0.37 kg/m²hr and negligible membrane mass transfer resistance for the organic species to produce selective evaporation membranes by grafting a molecular layer of terminally anchored poly (vinyl pyrrolidone) (PVP) or poly(vinyl acetate) (PVAc) polymer chains to an otherwise non-selective porous alumina membrane with 50Å average pore size. The majority of the proposed inorganic-polymer composite membranes for pervaporation have relied on physical attachment of the polymer to the inorganic substrate via solution casting [12], coating [13, 14], dip-coating a porous ceramic substrate followed by subsequent cross-linking [15], asymmetric incorporation of

inorganic materials as a powder in a polymer matrix [16], or pore-filling of a porous ceramic substrate with acrylamide, followed by cross-linking [17]. In contrast, ceramic-supported polymer membranes are created by terminally and covalently bonding a layer of polymer chains onto the surface of a porous ceramic support. The polymer surface chains are expected to have greater mobility than cross linked chains, yet the polymer phase is stable even when contacted by liquid mixtures in which the native polymer is completely soluble. In principle, the temperature stability of the composite stable polymer (CSP) membrane is limited only by the temperature at which the thermal degradation of the polymer backbone or side groups may occur. This membrane type is particularly attractive because a wide variety of monomers can be used in the graft polymerization reaction, thereby allowing one to tailor design such membranes for an array of different pervaporation applications. For example, Leger et al.

[18] Grafted preformed polydimethylsiloxane (PDMS) onto the surface of an alumina support membrane by contacting the support surfaces with heated silicone oil. The resulting pervaporation membrane was tested for the separation of aqueous solutions of 5 wt % 1-butanol and 10 wt % tetrahydrofuran (THF), resulting in a THF separation factor of 20 and 1-butanol separation factor of 20 for their respective solutions. Total permeation fluxes for the THF and 1-butanol aqueous solutions were 0.1 kg/m² hr and 0.31 kg/m² hr, respectively. The main advantage of chemical gels is the development of

considerably stronger bodies in comparison to the particle network of physical gel.

Finer particles must be dispersed in to organic material with a view to obtaining a more homogeneous composite by the application of alkoxide sol. [19, 20]. Where many authors has been studied the application of different binders to gave a homogeneous polymer solutions composite such as polyvinyl alcohol at different degree of polymerization in order to increase the flexibility of green gel composite or polymer dispersion latexes consist of small (30-500nm) polymer particles (acrylic) which dispersed in water [21-22]. In order to suitable application of the final gel composite products (AL₂O₃/PVA) composite gel in casting of complex shapes industrial material.

Experiment

Materials:

1. Alumina rout from natural sources in Iraq (bauxite ore) of average pore size 50 Å.
2. Polyvinyl alcohol for composite gel process (Fisher Scientific, Tustin, CA).
3. Sodium salt to distribute the alumina particles as Na₂CO₃ (Aldrich Chemical Co., Milwaukee, WI).
4. H₂SO₄ BDH Company of 99.9% conc.
5. Paraffin film for molding stage before testing the final gel composite.
6. Moulds from available metal compound such as chrome-nickel alloy at different shapes to suitability the test properties.

7. CH₃COOH organic acid reagents (Aldrich Chemical Co., Milwaukee, WI).

Procedures

The preparation of final composite gel is carried out by the following stages:

1. The preparation of raw refractory material: where the bauxite ore (rout of alumina) is collected milled (<25 μm) classified then calcinations at 850°C for 24 hours to stabilize its final properties before application it in the polymerization and preparation of gel composite product.
2. The preparation of polyvinyl sol. By hydrolysis stage (solution polymerization): This stage is carried out by dissolving different mass ratios of polyvinyl alcohol (PVA) in H₂O solvent as additive weight ratios (10, 20, 30, 40, and 50) wt% in order to give different degree of polymerization (0.5%wt of CH₃COOH catalyst was used to push this reaction forward). These polymer binder PVA has been quickly hydrolysis in a stirred glass tank reactor (CSTR) batch of 500ml volume at 200°C for 2 hours to gave approximate homogeneous transpierce sol. A different mass ratios of refractory material of alumina (<25μm) particle size are added as (5, 10, 15, 20, 25) wt% of Al₂O₃ respectively to polymerized PVA with a continuous stirring at 175 cycle/min for 4 hours at 350°C to give a homogeneous composite gel of Al₂O₃/PVA, a dispersion agent

of Na₂CO₃ was used to give a continuous homogeneous phase of composite gel Al₂O₃/PVA during the polymerization second stage and complete the reaction without any residue of active rout alumina as shown in table (1).

3. Finally the prepared composite gel are molded in a preparation mould surfaced with a paraffin films to easy dump the final composite gel product without any distortion and cracks on the specimen before any test a chivied .Then curing these molds were curing at 120°C for 2 hours to compatible and dry the final gel composite.

2. Properties of final gel composite products Al₂O₃/PVA.

1. Chemical resistance for chemical solution:

This test is carried out for a gel composite specimen at specified dimensions of (3 cm diameter, and 3mm thickness) soaked in chemical solutions (100%H₂O and 10% H₂SO₄) at 50°C for 5 days to check its weight loss every 24 hours respectively.

2. Thermal properties:

2.1. Thermal conductivity (K):

This test is carried out by the use of Lee-disk tester (three point bending tester) for a specimen at specified dimensions of (3 cm diameter and 3mm thickness) applied between two copper disk until reached to a thermal stability then a temperature reading are recorded as (T₁, T₂, T₃) .afterward using these two

equations to calculate a thermal coefficients (e and K) [24].

$$IV = \pi r^2 e (T_1 + T_3) + 2 \pi r e [d_1 T_1 + Ds (T_1 + T_2) + d_2 T_2 + d_3 T_3]$$

..... (1)

$$K [T_2 - T_1 / Ds] = e [T_1 + 2/r (d_1 + 1/2 Ds) T_1 + 1/r Ds T_2] \quad \dots (2)$$

Where:

e = loss of heat per m².

IV = rate of energy supplied.

d = thickness of disk m.

r = radius of disk m.

Ds= thickness of sample m.

T₁, T₂, T₃ = temperatures through of copper disks.

2.2. Thermal stability °C:

This test is carried out by the use of same dimensions of specimen (3 cm diameter and 3mm thickness), then accumulating exposure to an increasing in temperature at heating rate 30°C/min in a suitable furnace afterward checks its distortion property (change in dimensions) and finally records the distorted temperature in (°C) .

2.3. TG- analysis:

This test was carried out in a manual TG – system (in the industrial lab in chemical engineering department) to suit our study of weight loss for bulky samples of gel composite product. This thermogravimetric system applied the increasing of temperature for composite samples under a nitrogen flow rate (inert gas) at 0.35m³/hr where the sample was placed in a stainless steel basket of (10cm diameter. 30

cm length). The basket is hanging to the bottom of semi-micro-balance by 100 cm stainless steel wire as shown in Figure (1).

The temperature is measured at a specified point along the reactor of test (center wall) during constant time interval each (10 min) where both temperature and weight loss are recorded in order to investigate the TG-curve of find gel composite product.

3- Mechanical Properties:

3.1. Compression test (Mpa):

Compressive strength is the maximum stress that a rigid material with stand under longitudinal compression which is measured as force per initial cross-section area as. As the standard procedure in ASTM-D695 [25].

Compressive strength = compressive stress/ compressive strain (Mpa)(3)

3.2. Tensile Strength:

Tensile Strength was determined by Zweigle instrument as the procedure of standard ASTM-D-648[26].

At a constant test velocity applied at 0.5 cm/min until breaking occurs then recorded the tensile load finally.

Results and Discussion

- a) Use of a dispersion agent Na₂CO₃ to prevents any distortion in shape during the molding and handling steps. In addition leading to a close approximation of neighboring particles and give more pronounced effect on the attractive wander walls forces.

- b) The stability of characteristic properties of final gel composite product requires not less than 24 hours after curing and drying also before any test achieved.
- c) The additive of active rout refractory alumina is applied on the prepared PVA sol. in order to give a homogeneous gel composite without any residue in the bottom of dish.
- d) A calcinations of the active refractory rout alumina (bauxite ore) is achieved before any additive to stabilize its properties and remove any crystal moisture in the structure source material and gave along Al_2O_3 component pure characteristic properties of gel composite product Al_2O_3 /PVA.

Properties of gel composite products Al_2O_3 /PVA.:

1. Chemical resistance:

Different chemical solutions were used to study its effects on the final gel composite product Al_2O_3 /PVA, fig. (2) shows the effect of H_2O solution on the degree of swelling of final gel composite Al_2O_3 /PVA where the change in weight is increased with increasing of aging time from (24-120) hrs due to the reaction of OH groups in the structure of PVA with water and convert it to a soluble group (by eyes). Also this figure is stated the optimum mixing ratio from both PVA sol. To activate rout of Al_2O_3 of sample No. 3 of 30 PVA sol. /15 Al_2O_3 .

Fig. (3) indicate the effect of acidic solution 10% H_2SO_4 on the degree of swelling of final gel composite

product. Where the change in weight is increased with aging time from (24-120hrs.) at $50^\circ C$ due to the effectiveness of hydroxyl group (OH) , also the optimum mixing ratio that have less change in weight is sample No. 3 of (30 wt% PVA/15wt% Al_2O_3) because it's a stable sample and never change by any weathering conditions (conc. , time , temp.).

Fig. (4) shows a comparison for the double chemical solutions (100% H_2O and 10% H_2SO_4) on the chemical resistance (degree of swelling) and the change in weight for optimum mixing ratio, which prove that a less change in weight for samples exposures to moisture solution than acidic one due to normal effect of H_2O and acidic effect of alumina rout that will be appeared at increasing of Al_2O_3 ratios 20 and 25wt% respectively [8-23] see tables (2&3) all sample are stable after 50 hrs. .

2. Thermal Properties:

Fig. (5) shows the effect both polyvinyl alcohol binder solution and activated rout of Al_2O_3 (alumina) on the thermal conductivity of final gel composite product where a thermal conductivity coefficient (K) is decreased with increasing both PVA binder /and Al_2O_3 rout until reach optimum sample No.3 then give stability constant effect at an increasing in alumina rout due to a high thermal stability of alumina rout and complete polymerization reaction of composite gel at this ratio , also any additive on this ratio cause a precipitation residue and side reactions to the final gel [1,23] .

Fig. (6) shows the thermal stability of binder PVA and active

Al₂O₃ rout. The thermal stability is increased with increasing of Al₂O₃ rout until reach optimum additive of 30%PVA/15%Al₂O₃ then reached constant stage due to a constant effect of final gel and complete reaction at this optimum ratio , also any excess of this ratio cause a residue precipitation unreacted substance of alumina [1,23].

Fig. (7) is studied the TG-analysis curve to certain the thermal stability results where the optimum mixing ratio of (30%PVA/15%Al₂O₃) gives high stability and less loss in weight than other base polyvinyl alcohol due to high thermal stability of activated rout Al₂O₃ than other base of PVA and complete of polymerization reaction[1,23] see tables (2&3) .

3. Mechanical properties:

Fig. (8) indicate the effect of PVA/AL₂O₃ on the resistance to compressive strength load , where this strength is increased with increasing the additive of activate rout AL₂O₃ until reach optimum additive (sample No.3) then become constant due to complete reaction at this additive and any increasing cause unreacted precipitate residue and cause a drop in mechanical properties[3,27].And fig. (9) shows the effect of both PVA/AL₂O₃ on the tensile strength of final composite gel, where The tensile strength is increased at increasing of activate rout AL₂O₃ until reach optimum ratio (sample No. 3) then become constant due to a complete reaction at this ratio, also any excess of active route

cause a precipitation residue and drop in mechanical properties [13, 27] see tables (2&3).

Conclusions

1. The size for PVA alone increases to 2.3 times of its original value by the thermal conductivity. However that of PVA in the composite increases slightly to 1.1 times by similar treatment.
2. The crystallization not proceeds in the composite since a movement of the molecular chain of PVA should be restricted by the interaction with alumina which is very strong judging from the sorption and swelling.
3. The maximum stresses (tensile strength and compression strength) (2.4, 1.1) than the standard (1.0, 0.5) appeared in optimum mixing ratio of sample No. 3 of (30%PVA/15% Al₂O₃).
4. The minimum change in weight is for optimum mixing ratio sample No. 3 of (30%PVA/15% Al₂O₃).
5. High stability appeared for composite gel product 30%PVA/15% Al₂O₃ (99%) than base polymer PVA(42%).
6. From the above test results it could conclude that this type of composite (product) is used for the preparation of complex shape moulds in the industrial applications.

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Table (1) shows the mixing ratio of active route (Al₂O₃) to the base polyvinyl alcohol.

Sample No.	Al ₂ O ₃ wt. %	PVOH* wt. %	H ₂ O wt. %
1	5	10	85
2	10	20	70
3	15	30	55
4	20	40	40
5	25	50	25

* Polyvinyl alcohol

Table (2) indicates the comparison between properties of standard PVOH to composite gel Al₂O₃ / PVOH optimum one.

Sample type	Chemical resistance*		Thermal conductivity K (w/m.°C)	Thermal stability		Compression test (MPa)	Tensile strength test (MPa)
	H ₂ O wt. %	H ₂ SO ₄ wt. %		°C	Loss in wt. %		
PVOH (standard)	2.0	2.4	0.015	200	42 %	0.5	1.0
Optimum ratio Al ₂ O ₃ (15%)/PVOH(30%)	2.3	2.4	0.0075	325	99 %	1.1	2.4

* Chemical resistance: indicate the absorption of moisture and other solutions at sever conditions for time and temperatures.

Table (3) the improvement in properties (%) between standard and optimum mixing ratio (15% Al₂O₃ / 30% PVOH) wt.

Sample type	Chemical resistance		Thermal conductivity	Thermal stability		Compression test MPa	Tensile strength test MPa
	H ₂ O %	H ₂ SO ₄ %		°C	Loss in wt. %		
PVOH (standard)	2.0		2.4	0.015		200	42%
Optimum ratio	30%	constant	50%	62.5%	57.5%	54.5%	58.55

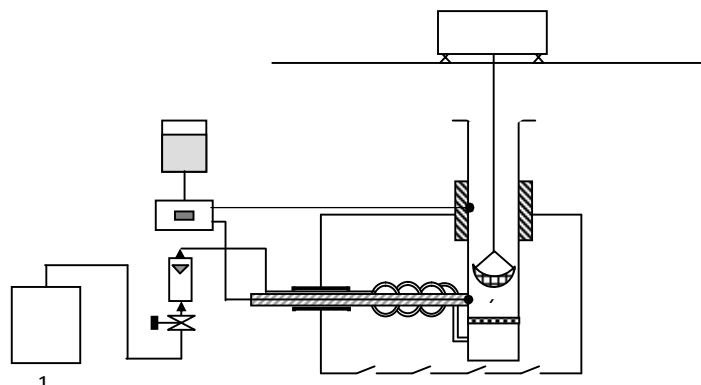


Figure (1) Experimental apparatus for TG-analysis.

1- Gas source with regulator .2- gas rotameter.3- stainless steel reactor. 4- Gas distributor. 5- Electrical furnace .6- stainless steel basket .7- sensitive balance.8- selector switch .9-digital thermometer .10- stainless steel coil. 11- Stainless steel wire.

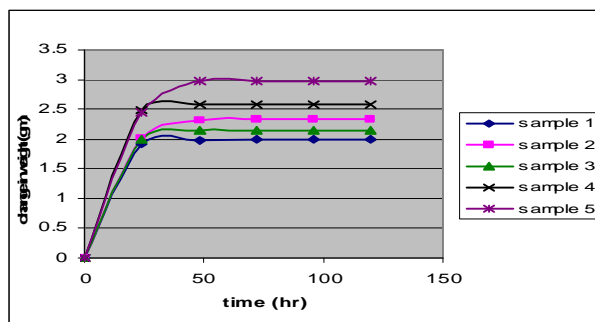


Figure (2) shows the chemical resistance in moisture container 100% H₂O for 5 days and 50°C.

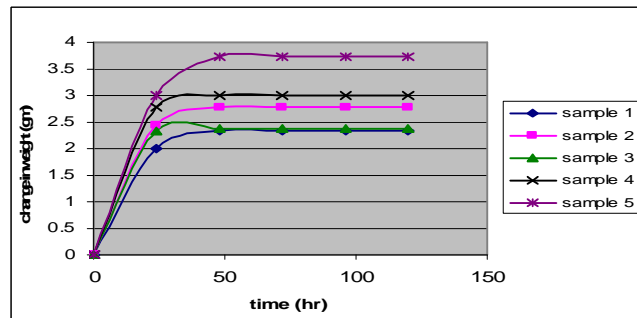


Figure (3) shows the chemical resistance for all samples in acidic solution 10% H₂SO₄ for 5 days at 50°C.

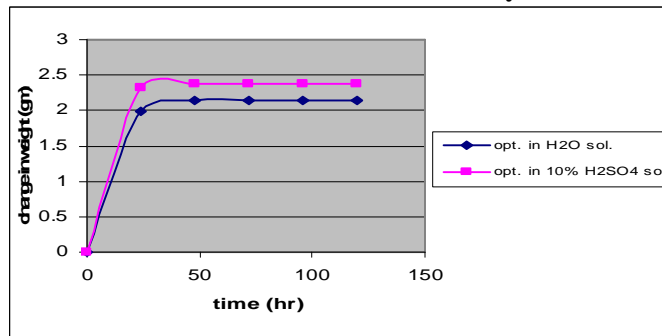


Figure (4) indicate comparison between two chemical solutions results for optimum samples for chemical resistance property.

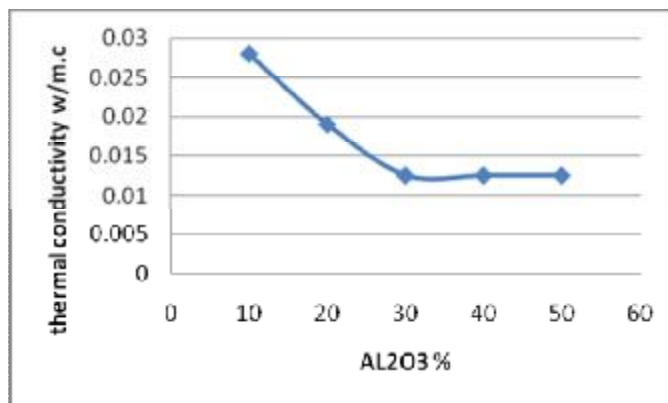


Figure (5) shows the effect of Al₂O₃ additive on the thermal conductivity coefficient of composite prepared gel.

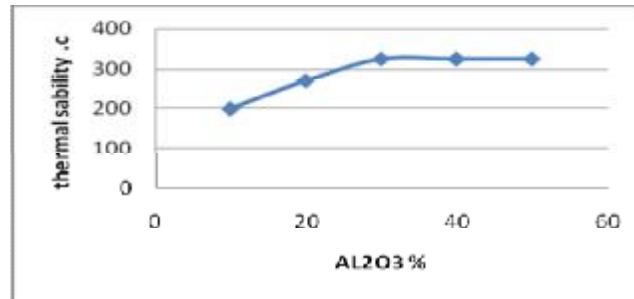


Figure (6) shows the effect of Al₂O₃ additives on the thermal stability of composite prepared gel.

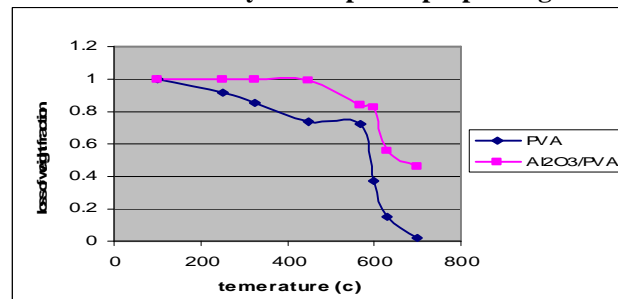


Figure (7) shows the TG-curve for both base PVA and composite prepared gel Al₂O₃ /PVA.

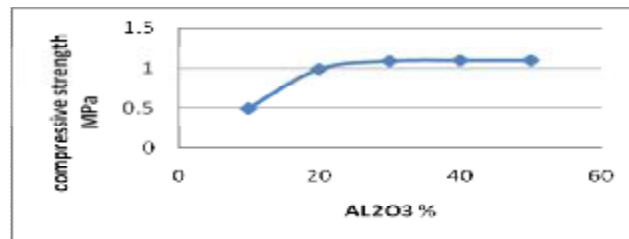


Figure (8) shows the effect of Al₂O₃ additives on the compressive strength of composite gel prepared.

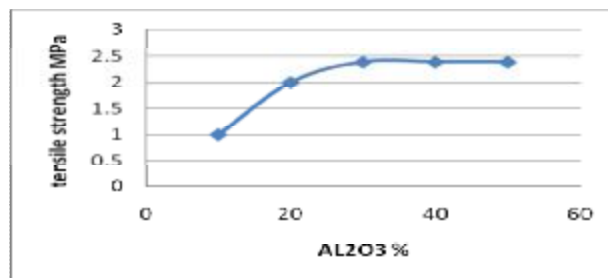


Figure (9) shows the effect of Al₂O₃ additives on the tensile strength property of composite gel prepared.