Fabrication of Hydrophobic Nanocomposites Coating Using Electrospinning Technique For Various Substrate

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Abstract. Hydrophobic coating were fabricated using electrospinning technique on different substrates (glass, ceramic and metal) , polymer solutions of (Si) were used in different range of composition for each solutions, also nano Al₂O₃ and nano TiO₂ were used to fabricate nanocomposites. Contact angle, surface tension, viscosity, X-ray diffraction and FTIR were calculated for all specimens. (20%Si) owned a larger contact angle about (110.173°) for metal substrate and show a hydrophobicity for ceramic and glass substrate after coating with (20%Si). SEM showed the morphology of the surfaces and show that specimens has good surface morphology after coating with (20%Si/Al₂O₃) and (20%Si/TiO₂). All results were discussed.

Keywords : (Si) Silicone Rubber R.T.V. , (Th) Thinner , (TiO₂) Nano Titanium dioxide , (Al₂O₃) Nano Alumina, (CA) Contact angle , (SEM) Scanning Electron Microscope

1. Introduction

Recently, interested of engineering and nanotechnology increased to the hydrophobic materials and coatings according to their requirements to obtain material with the requested properties which are better than raw materials. This research focused on studies the hydrophobic coating to achieve materials with special surface morphology by calculating wettability of coated surfaces (ceramic, glass and metal) substrate to find out the hydrophobicity and characteristics of coated surfaces, these substrate were picked up because they are very common in use to daily people life and industrial manufacturing which control an special role around this research. Also tested to Scanning electron microscope (to show the morphology texture of coated substrate).

Hydrophobic coating, is the resistance of material surface to the water and according to the contact angle test, hydrophobic must have an angle acceded more than 90° which between (90° - 150°). There are different types of hydrophobic substrate in nature, these examples has hydrophobicity and low adhesions properties. These special characteristics showed a great consideration by science researchers that involving (self-cleaning, anti-corrosion, anti-wetting ..etc.). So this article specialized on fabricate hydrophobic coating to obtain what almost in nature types and achieve these special requested properties[1].

Electrospinning is one of the most practical and better method which become familiar and popular in the last years and interested grown up towards nanotechnology because of the production of nanofibers polymer with diameter (2nm - 5μm). Generally, electrospinning process contains syringe pump, high voltage source and collector, the polymer solution place inside syringe pump and boosted in constant flow and the needle connected to high voltage source in range (3KV - 30KV)[2].
Parameters of electrospinning process are the scale of the calculated material from electrospinning process that affect on the fibers properties. There are many types of electrospinning parameters (Solution parameters, Instrumental parameters and Ambient or Environmental parameters) [3].

Viscosity is very important in electrospinning technique which determine by the add of polymer concentration to the solutions, factors effect on viscosity is molecular weight and the concentration of the polymer.

Surface tension is also one of the substantial agent effect on electrospinning operation which affect by the composition of polymers solution and this will outcome with changing the surface tension. When there is high amount ratio of free solvent molecules, molecules mixed to each other and form beads [4].

Contact angle, define the wettability of the substrate which are very useful in this article which involve the perusal of contact angle test as primary data, and calculated the degree of wettability when the water drop touch surface that been coated. Contact angle split to many parts and every sections has values of wettability range and resistance of the substrate to the water drop, samples in this research will calculated by this test to define the wettability of the substrate that fabricate by electrospinning process, sections of CA are (figure (1) shows CA range) [5]:

![Figure 1. Contact Angle Range.](image)

2. Experimental Part

2.1 Materials Used

1. Silicon Rubber R.T.V.

2. Thinner Solution (Sun Shine Futureline).

3. Nano titanium dioxide (TiO₂) nano particle with diameter (20nm - 40nm) from micxy reagent company.

4. Nano Alumina (Al₂O₃) nano particle with diameter (20nm - 30nm), purity (99.99%).
2.2 Preparation of Samples

Substrate used are (ceramic, glass and metal) each of these surface has cleaned with distilled water and alcohol for 5 min then dried in oven at temperature of 35-40°C to achieve a smooth substrate without infection and scotch to the surface [6]. Figure (3) shows samples prepared before been coated.

![Figure 3. Sample Preparation (Ceramic, Glass and Metal) before coating.](image)

Preparation of polymer solution is done by weighed silicon rubber R.T.V. in various wt% composition and dissolve it with thinner solution, the percentage of (Si/Th) solution prepared in this research are in range of (10%, 15%, and 20%) of Si, and placed on a magnetic stirrer for 6 hours to make an homogenous polymer solution, and the best contact angle of (Si/Th) solution coated to metal substrate is (20% Si/Th) was depended in this article because it shows the larger CA over other percent which shows about (110.173°) degree and depending on this percent another solution prepared by addition of nano Al2O3 and nano TiO2. Figure (4) shows various polymer solution of (Si/Th), (20% Si/Th/Al2O3) and (20% Si/Th/TiO2) [7].

![Figure 4. Polymer Solutions of (Si/Th), (20% Si/Th/Al2O3) and (20% Si/Th/TiO2).](image)

For metal sample is coated with (Si/Th) solution with different wt% composition of (10%, 15%, and 20%), and resultant with hydrophobicity properties after been coated and tested to CA test, that provide good enhancement to the surface of the specimen, figure (5) shows metal samples coated with various composition of (Si/Th) solution:
Figure 5. Metal samples coated with various composition of (Si/Th) solutions.

(20% Si/Th) shows larger CA to other coated samples and depending on this percent, a solution of (20% Si/Th/Al₂O₃) and (20% Si/Th/TiO₂) also prepared to fabricate nanocomposite coating by adding a known amount of each nanoparticle used that mentioned, figure (6) shows metal samples coated with (20% Si/Th/Al₂O₃) and (20% Si/Th/TiO₂) solution:

Figure 6. Metal samples coated with (20% Si/Th/Al₂O₃) and (20% Si/Th/TiO₂) solution.

For glass sample is coated with (Si/Th) solution with different wt% composition of (10%, 15% and 20%), and resultant with hydrophobicity characteristic after being coated and tested to CA test, which provide good enhancement to the surface of the sample, figure (7) shows glass samples coated with various composition of (Si/Th) solution:

Figure 7. Glass samples coated with various composition of (Si/Th) solution.
For ceramic sample is coated with (20% Si/Th) solution and resultant with great enhancement to hydrophobicity characteristic from (0°) of CA which mean superhydrophilic to hydrophobic surface of (90°) of CA test after been, figure (8) shows ceramic sample coated with (20% Si/Th) solution:

![Figure 8. Ceramic sample coated with (20% Si/Th) solution](image)

Installation of electrospinning operation should be set up with advance handle person and care should take. Generally, the operation of electrospinning has syringe pump, high voltage source and collector, initially, this process contains of 3 electrode, the polymer solution that prepared previously explained, placed inside syringe pump, one of the electrode is connected to the needle of the syringe pump, the needle diameter is connected with high voltage source (6.5KV used in this research), the syringe used is 3ml size, which passing in static flow rate. other electrode is connected to the plate that hold samples substrate on it which named collector and the third electrode is connected to earthen to prevent electrical fault shock [8].

When an electrical current is passing to the needle of the syringe pump it generate electro static force that changing the surface tension of (Si/Th) solution and deform the shape of spray on collector plate and when the voltage increased the change in the shape of the spraying increased, when the voltage stabilized it work on evaporate the solute and coating fabricated by this process only stay on the surface of the sample [2-3]. Figure (9) show electrospinning technique method preparation and set up as explained previous section.

![Figure 9. The Electrospinning Technique Method Preparation and Set-up.](image)

3. Results and Discussion

Table (1) shows the result of prepared (Si/Th) solutions after tested to viscosity and surface tension tests. For viscosity show increasing with ascending the percent of Si that mixed with Th solute. By ascending viscosity of the (Si/Th) solution much higher with high molecular weight or ascending the Si concentration in the solution, then a mixture of aggregate and beads will be obtained. Figure (10) shows relation between viscosity and surface tension [9].
Table 1. Viscosity and surface tension of (Si/Th) solutions.

| Polymer Solution | Viscosity (m²·sec⁻¹) | Surface Tension (N·m⁻²) |
|------------------|----------------------|-------------------------|
| (10%Si/Th)       | 12.15                | 10.55                   |
| (15%Si/Th)       | 13.68                | 12.9                    |
| (20%Si/Th)       | 19.06                | 17.38                   |

Figure 10. Relation between viscosity and surface tension.

Contact angle has tested for all samples that prepared before and after coating with electrospinning process. For metal substrate CA test shows good enhancement from hydrophilic material to hydrophobic material, (10%, 15% and 20%) of (Si/Th) solutions are coated to metal samples and the larger CA was the specimen of (20%Si/Th) which about (110.173°) degree. Figure (11) shows CA result for metal surface coated with different composition of (Si/Th) solutions.
For glass substrate CA test shows good enhancement from hydrophilic material to hydrophobic material, (10%, 15% and 20%) of (Si/Th) solutions are coated to glass samples and the larger CA was the specimen of (15%Si/Th) which about (107.491°) degree. Figure (12) shows CA result for glass surface coated with different composition of (Si/Th) solutions.

For ceramic substrate CA test shows great enhancement from superhydrophilic material to hydrophobic material, For ceramic (None) substrate shows (0°) CA because of high porosity of the surface which comes out as a superhydrophilic material, (20%Si/Th) solution coated to ceramic sample and shows a hydrophobicity to the surface of the sample. Figure (13) shows CA result for ceramic surface coated with different composition of (Si/Th) solutions [10].
Figure 13. CA for ceramic specimens coated with (20% Si/Th) solution. A. (None) CA=0°, B. (20% Si/Th) CA=90.667°.

Table (2) shows the balance of CA test for left CA, right CA and average of CA for all specimens coated with (Si/Th) Solutions.

| Polymer Solution | Contact Angle Left (CA_L) | Contact Angle Right (CA_R) | Contact Angle Average (CA_AV) |
|------------------|---------------------------|----------------------------|-------------------------------|
| (None) - Metal   | 60.493                    | 60.493                     | 60.493                        |
| (10% Si/Th) - Metal | 95.624                 | 95.624                     | 95.624                        |
| (15% Si/Th) - Metal | 99.314                 | 100.386                    | 99.850                        |
| (20% Si/Th) - Metal | 109.158                | 111.189                    | 110.173                       |
| (None) - Glass   | 47.647                    | 47.647                     | 47.647                        |
| (10% Si/Th) - Glass | 95.912               | 90.544                     | 93.228                        |
| (15% Si/Th) - Glass | 107.491               | 107.491                    | 107.491                       |
| (20% Si/Th) - Glass | 100.507               | 100.507                    | 100.507                       |
| (None) - Ceramic | 0                        | 0                          | 0                             |
| (20% Si/Th) - Ceramic | 92.843              | 88.490                     | 90.667                        |

As mentioned in previously section metal samples of (None), (20% Si/Th), (20% Si/Th/TiO₂) and (20% Si/Th/Al₂O₃), that owns best contact angle as compared to other specimens has also tested to X-ray diffraction to show the structure of fabricated materials or coating. Figure (14) show x-ray diffraction pattern for mentioned specimens and show that specimen without coating has less peak while sample of (20% Si/Th) shows large peaks and also show new three different range of peaks and considered theta steady at 3 position which is about (64.9516°), (78.0959°) and (44.5536°). Other specimen coated with (20% Si/Th/TiO₂) and (20% Si/Th/Al₂O₃) shows new and different range of peaks, these two sample with nano
particle coated substrate also owns much larger peaks and higher rate of intensity as compared to (None) and (20%Si/Th) specimens.

Other test was examined for materials used of (Si R.T.V.) , (Nano-TiO₂ Particles) and (Nano-Al₂O₃ Particles) by collecting of remains in coating of (20%Si/Th) , (20%Si/Th/TiO₂) and (20%Si/Th/Al₂O₃) at up of the surface and scratch it to have amount of these powder and test it to FTIR spectrum test, figure (15) shows the FTIR spectra of mentioned materials , for (20%Si/Th) FTIR spectra showed some aggregate of peaks in range of 3100 to 3600 cm⁻¹ that have unstable range of multiple peaks confirming that the produced coating is of crystalline form, for (20%Si/Th/Al₂O₃) shows board peak in range of 500-900 cm⁻¹ , and for (20%Si/Th/TiO₂) the broad band centered at 500-600 cm⁻¹ is assigned to the bending vibration bond in TiO₂ lattice , the broad band centered at 3600-3400 cm⁻¹ is assigned to the intermolecular interaction of Si group with metal substrate.

Metal Samples of (20%Si/Th) , (20%Si/Th/TiO₂) and (20%Si/Th/Al₂O₃), that owns best contact angle as compared to other specimens has tested to scanning electron microscope test (SEM) to see the morphology of the surfaces, and show that specimen of (20%Si/Th/Al₂O₃) have higher amount of beads and this due to the moderate viscosity or increasing of Si concentration in the Th solution, then a mixture of aggregate and beads will be obtained, and due to unbalanced applied voltages in range of (6.5KV - 7.8KV) also the use of roller tube collector to obtain alignment coating, as in figure (17).

While in (20%Si/Th) show beads and defect and this forms because (Si/Th) solution has low viscosity also not need a matrix because already adhesion with surface so it appears in this way as in the figure (18), and for (20%Si/Th/TiO₂) shows a great bonding because of the presence of TiO₂ nanoparticles which owns very high surface area that helps to bond together with less beads. as in the figure (19) [11 - 12].
Figure 14. X-Ray Diffraction Pattern for Metal substrate.

Figure 15. FTIR Spectrum for Collecting Remains of Coating At Metal Substrate.
Figure 16. SEM images for (20%Si/Th/Al<sub>2</sub>O<sub>3</sub>) - Metal substrate.

Figure 17. SEM images for (20%Si/Th) - Metal substrate.

Figure 18. SEM images for (20%Si/Th/TiO<sub>2</sub>) - Metal substrate.
4. Conclusions :

- (20%Si/Th) for metal substrate owns the higher contact angle about (110.173°) as compared to other (Si/Th) coated samples. (15%Si/Th) for glass substrate owns the higher contact angle about (107.491°) as compared to other (Si/Th) coated samples.
- (20%Si/Th) has higher amount of beads due to the viscosity of (Si/Th) solution.
- (20%Si/Th) has higher viscosity and surface tension.
- Both metal and glass substrate obtains good enhancement from hydrophilic to hydrophobic material.
- Excellent enhancement for (ceramic substrate) after coating by electrospinning process with (20%Si/Th) solution and contact angle changed from superhydrophilic (0°) to a hydrophobic (90.667°) , and the ceramic surface become repels water instead of absorb water.

5. References:

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