Fabrication Of TiO_2 , V_2O_5 Thin Film (Super Hydrophobic Surface)By Powder Coating Technique

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ABSTRACT

In this research, deposition of titanium oxide (TiO_2) and vanadium oxide (V_2O_5) thin film in different mixing percentage (0, 25 ,50, 75 and100)% on the substrate of glass .The coating thickness was (50 nm).

In this research contact angle was measured and the effect of weather conditions. Results showed that the value of the contact angle of the prepared films reached its highest value at 50% (TiO_2+V_2O_5) was 160º.

The results showed that the optical transmittance of TiO_2 and V_2O_5 thin film decrease with increasing the deposition angle and decrease with increasing V_2O_5 proportion.

KEY WORDS :TiO_2,V_2O_5,thin film ,contact angle, optical transmittance, super hydrophobic surface.

تصنيع أغشية رقيقة من TiO_2 , V_2O_5 (سطح فائق الرفض للماء) بتقنية طلاء المسحوق

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الخلاصة:

في هذا البحث، تم ترسيب أوكسيد التيتانيوم (**TiO_2**) وأكسيد الفناديوم (**V_2O_5**) بشكل غشاء رقيق عند نسبة خلط مختلفة (**0** و**25** و **50** و **75** و **100**)% منها على قاعدة من الزجاج. وبسمك طلاء (**50** نانومتر).

في هذا البحث تم قياس زاوية الاتصال وتأثير الظروف الجوية . وأظهرت النتائج أن قيمة زاوية الاتصال من الأفلام المعدة بلغت أعلى قيمة لها عند نسبة خلط **50**٪(**TiO_2**، **V_2O_5**) و كانت (**160**°) .

وقد تم فحص النفاذية البصرية؛ وأظهرت النتائج أن النفاذية الغشاء الرقيق لكل من **TiO_2** و **V_2O_5** انخفضت مع زيادة زاوية الترسب ومع زيادة نسبة **V_2O_5** في الخليط.

الكلمات الرئيسة: TiO_2 ، V_2O_5، غشاء رقيق، زاوية الاتصال، النفاذية البصرية، سطح فائق الرفض للماء.

1. INTRODUCTION

Recently titanium oxide (TiO_2) vanadium oxide V_2O_5 ultra thin films have been investigated with regards to their remarkable optical, electrical and photo electrochemical properties.

A number of methods have been employed to fabricate thin films, including e-beam evaporation ,M.Z. OBIDA, 2005, sputtering, M. N. ESFAHANI 2008, chemical vapor deposition S.H. LEE et.al ,2001and sol-gel process , J. YU, X. ZHAO, 2001, and Q. ZHAO. Among these methods the sol-gel process is one of the most appropriate techniques to prepare thin film.

Application of (TiO_2 ,V_2O_5) like microfiltration media properties, catalytic reactors, cathodic protection, orthopedics. Contemporary orthopedics commonly uses various types of implants which replace damaged or malfunctioning parts of the osteoarticular system. The implants are manufactured using a number of construction materials fulfilling specific requirements. To numerous metallic materials belong stainless steel and titanium alloys, W.A. Seattle, 2006.

The materials used for the implants working for a long time in a living organism environment ought to be bio-acceptable, resistant to the influence of the tissue environment, and compatible biochemically. Also the implant surfaces are known to be very important, because their chemical, biomechanical, and topographic features influence the behavior of cells during the initial stage of the implant integration with the surrounding tissues, ultimately determining the speed and the quality of new tissue formation M.Z. OBIDA, M. N. ESFAHANI.

Physical properties of metallic materials which accelerate the development of bone–implant interactions can be improved by various techniques of surface film engineering, e.g. by the deposition of ultra thin oxide films by the sol–gel method, S.H. LEE et.al.

The main purpose of the present paper is fabrication of (TiO_2 ,V_2O_5) ultra thin films deposited on the surfaces of glass from its powders.

2. EXPERIMENTAL WORK:

This work includes many steps;

A- THE MATERIALS USED FOR THE PREPARATION OF THIN FILMS:-

The materials were used in this work to fabricate thin film are TiO_2 and V_2O_5 powders. These powders are prepared in sequent process is mentioned below:

Sieving process, milling process, and sieving process, particle size measurement process, preparation of materials ratio.

Sieving process was carried out in Material Engineering Department /University of Technology by the equipment device. Where using sieves measurements (93, 75.53, 38, 25) µm.

Milling process were done in Material Engineering Department /University of Technology by the equipment device, this process in 3 hour for each powder. Ball mill model 9 variable speed.

Particle size test was carried out in Advanced Materials Research Center at the Technology and Science Ministry. Specification of the device are (SALD-2101) laser diffraction particle size analyzer shimadzu. The results of the powder that was used in the research are as follows: V_2O_5=0.421 µm , TiO_2 = 0.390µm as in **fig (1)** and **(2)**.

Particle of powder materials have been weighed according to the selected ratio to prepare batches for the spray process by using balance device type Denver max weight is (210) g. This method are described in table (1).

B-SYSTEM PREPARATION:-

The system which is used for preparation of thin films by powder deposition process indicated in **fig (3)**.

This systems consists from a-nozzle b- compressor device c- electric heater d-flow meter e-beaker g –connection f- temperature measuring device remotely.

C- SUBSTRATE PREPARATION:-

All glass sheets were investigated in this work as substrate for preparation of surface in standard dimension as (5x2x0.2) cm and purity (99.99%). Cleaning process for the glass substrates was done by ethanol alcohol for 10 min , M. Mahdi1, 2009, rinse with distilled water, drying in air. After this operation the samples were putted and fixed on the electric heater with control temperature.

D- SPRAYING PROCESS:-

Pre chamber of the nozzle is designed as a unit for mixing carrier gas and for gas-powder, which moves down from the powder feeder to pre chamber. Low velocity gas-powder mixture entrances into pre chamber. Low-velocity gas- powder mixture moves from the feeder into pre chamber under high pressure (value of static pressure in powder mixture must be higher than pressure of carrier gas). That is why powder feeder must be designed considering high level of pressure inside. Pre chamber is connected to nozzle. Gas which we used in this work is air. Flow gas determine is (2.5 l/min) , at pressure (7) bar ,temperature is (25) C°. In the spray, unit powder is accelerated and heated by gas flow. After leaving the nozzle, particles interact with substrate and create a coating.

e- ANNEALING : was done at (350)C° for (one hour) in furnace type German origin (Nabertherm), maximum temperature up to 1100C° .

f- TESTING **:**

1- CONTACT ANGLE TEST:- Sample has been used to measure the contact angle for the droplet. According to the specification ASTM No (813-1990), ASTM,1988.

In this test, it is used the following items:-

a - rule b - camera type and strength enlarge(16 M pixel) to take pictures of the drop for each sample c - camera holder d - protractor e - light source f - needle or tube lattice equipped with a drop of water g - stopwatch h - thermometer and humidity k – computer. After that, contact angle can be plotted for each drop. Measure the angle from the right equal measure from the left. Samples that were used are st.st. Which has been stored in closed containers in order to keep this coat and uses gloves or carry special forceps.

Contact angle e apparent are shown in **fig(4)**, droplet is shown in **fig(5)**.

2- UV SPECTROMETER

TiO_2 and V_2O_5 thin films on glass substrate were irradiated by UV-VIS spectrophotometer.The device to measure the optical transmission of film is shown in figure (6). we were recorded the optical absorption and transmission for glass samples by UV-VIS spectrophotometer. From these it can find properties of the coating, thickness accurately by the equations (1) and (2) , M. Al-Mudhaffer, 2010.

$$\mathbf{r = (n-1)^2/(n+1)^2} \qquad \mathbf{(1)}$$

n : refractive index(dimensionless)

$$\mathbf{t = (1-r^2) * e^{-\alpha t}} \qquad \mathbf{(2)}$$

t: optical transmittance (percentage)

α: absorption coefficient(cm^{-1})

t: coat thickness (µm)

This device is available in nanotechnology and advanced materials research center / the university of technology with range (200-1200) nm.

3. RESULTS AND DISCUSSION

Fig(7) shows the contact angles of all the film which have been prepared at different deposition angles before exposure to Iraqi weather. In this figure note that component on the horizontal axis powder mixture concentration ratios (wt%) and the values of the contact angles in degrees on the vertical axis.

a-EFFECT OF COMPOSITION:-

The change ratios of powders included in the mixture, which was deposited affect on the value of the contact angle on the one hand. This figure represents a pre-exposure to weather conditions. The value of the contact angle of the titanium oxide reached almost 120º, and almost rise to 160º , X.Ding and Z. Wang, 2011.

b-Effect of deposition angle:

In the same figure an increasing the angle of deposition lead to increase the contact angle at all mixture concentration. At change of the deposition angle and this corresponds with the previous research, reaching to 151.5º, C. Xue and E.Denver, 2008.

Either in the case of vanadium oxide, it find that the value rise and reached 160º also with increasing deposition angle. and can conclude that angle deposition played a key role in changing the coat condition from hydrophobic to super hydrophobic and this value is identical with the reference ,K. Senthila and M. Kanehira, Z. Zhang, 2011.

Fig(8) illustrates change in contact angle after exposure to Iraqi weather condition during four months full of all samples. In this figure it noticed that the value of the change was few, as happened simple change in contact angle value and nearly all samples after four months full due to the influence of moisture and this corresponds to the reference , C.V. Ramana et.al, 2004.

In **Fig (9)** contact angle was taken per month for four months and four samples that showed the highest value contact angle through the first examination in **Fig(8)** with concentration (50% TiO_2,50%V_2O_5) and then examined for each month separately. Results also showed simple decrease in the values of the contact angle because increased humidity, which corresponds to (any moisture ratios) months installed in the same **Fig (9)**, because the increased humidity caused an increase in the area of exposure which cause that decreased M.Z. OBIDA et.al., 2005, C. Xue and E.Denver, 2008.

Optical transmittance have many application varied. Because it's important to have the coat or thin film high percentage of transparency in the area over the visible spectrometer. Therefore be appropriate for the application , C.V. Ramana et.al.

Fig (10:a) represents transmittance value of 100% TiO_2 and0% V_2O_5 and note that with increasing angle low transmittance because decrease case irregular film, which we observed in the images imaging microscopy in the preceding paragraph. The same think is true for the other thin films figures (10: b ,c ,d , e) that represent mixtures. Including all note on the whole low transmittance with increasing angle deposition, W. Gulbinski et.al, 2003.

4. CONCLUSIONS:

a-Real contact angle that have been measured (155º). That means the film was super hydrophobic especially at deposition angle of 45º.

b- It is clear that the optical transmittance of TiO_2 and V_2O_5 thin films decrease with increasing the deposition angle and decrease with increasing V_2O_5 proportion .Where it was in the case of pure titanium dioxide (82%) and decrease to (40%), maximum transmittance is (95%).

c-The contact angle measurements have highest value for mixture (50% TiO_2, 50%V_2O_5) increased with high angular deposition hitting (160º) at deposition angle (45º).

d- The influence of weather conditions was limited at this deposition angle (45º) for mixture (50% TiO_2, 50%V_2O_5) after exposure to four months consecutive full of Iraqi weather conditions.

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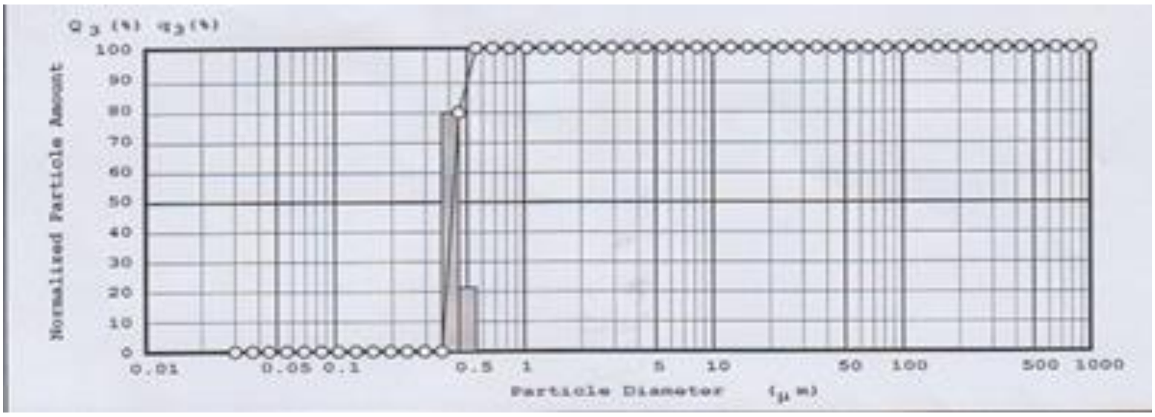


Figure (1) Particle size for V_2O_5.

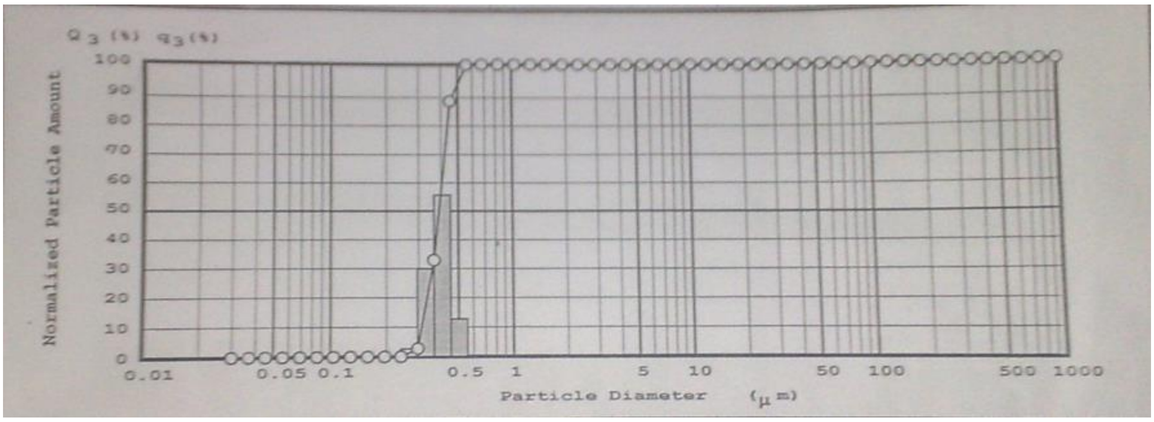


Figure (2) Particle size for TiO_2.

Table 1. Ratio of mixing powder materials according to weight percentage.

Sample symbol	A	B	C	D	E
Composition	100%TiO_2+ 0%V_2O_5	75%TiO_2+ 25%V_2O_5	50%TiO_2+ 50%V_2O_5	25%TiO_2+ 75%V_2O_5	0%TiO_+ 100%V_2O_5

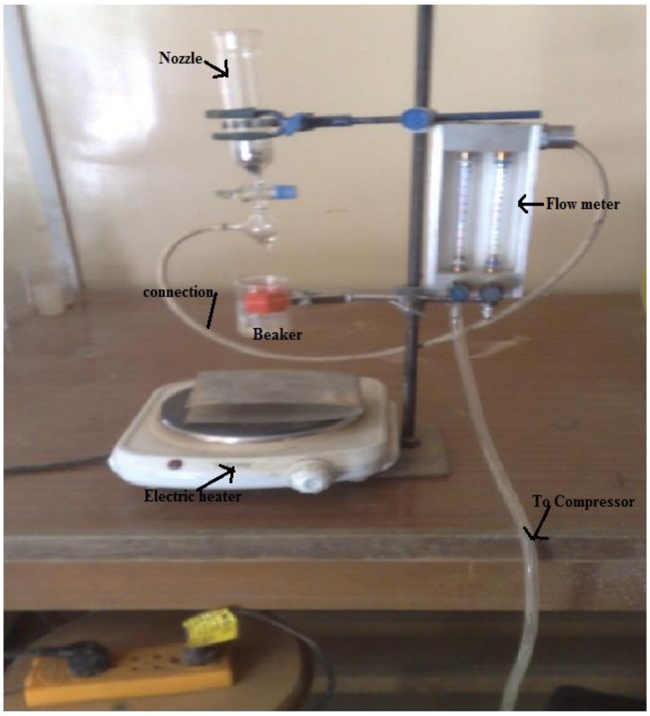


Figure 3. System for process

Figure 4. Contact angle apparatus

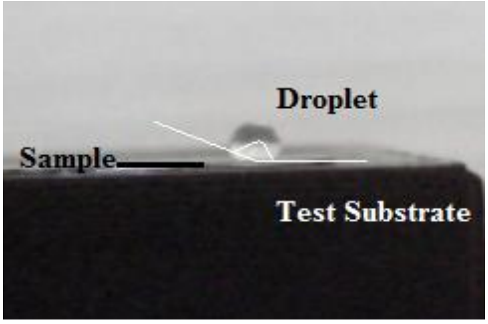


Figure 5. Droplet on the sample Figure 6. UV-VIS spectrometer device

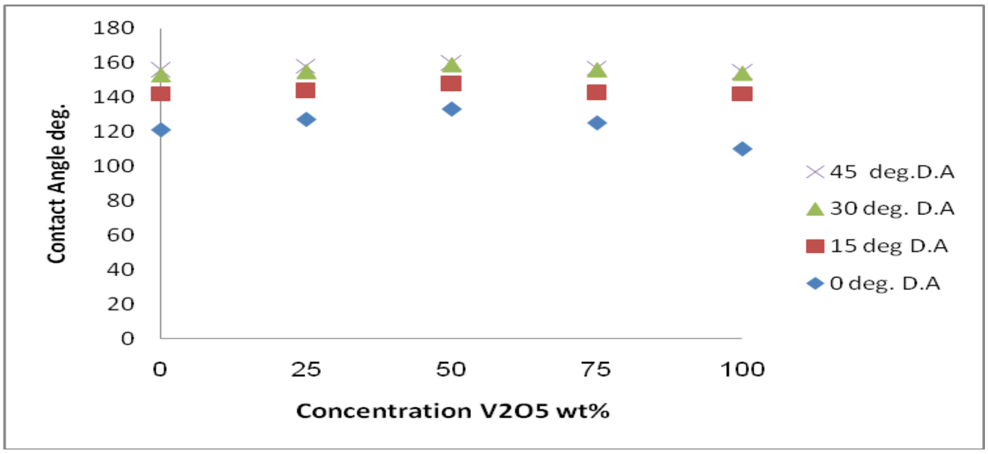


Figure. 7. Effect of V_2O_5 wt% on contact angle results before exposure to weathering and deposition angle.

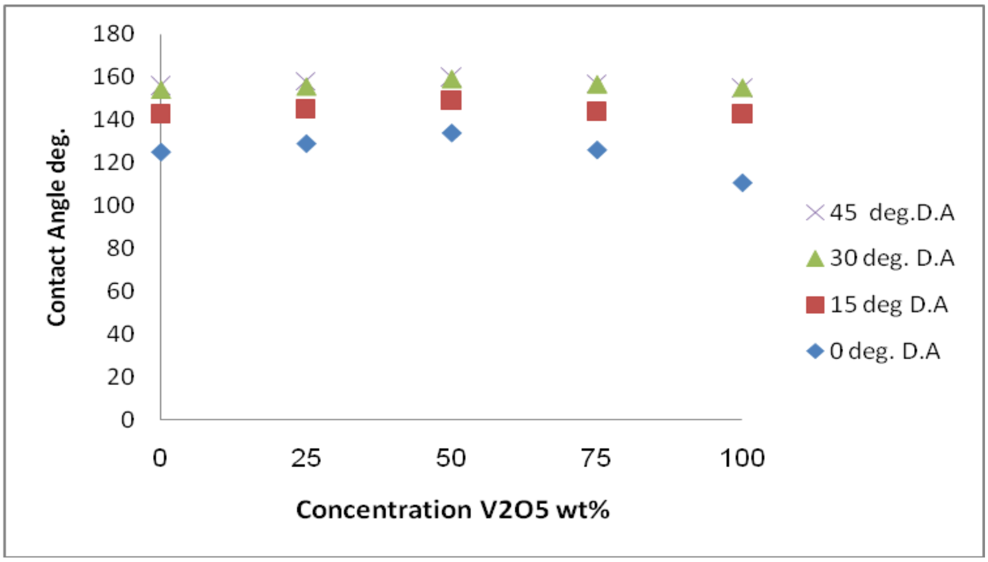


Figure 8. Effect of V_2O_5 wt% on contact angle results before exposure to weathering and deposition angle.

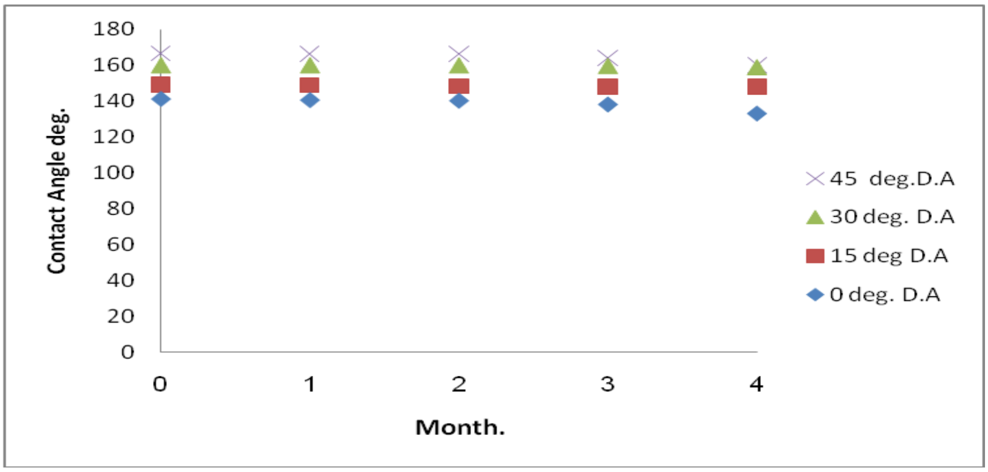
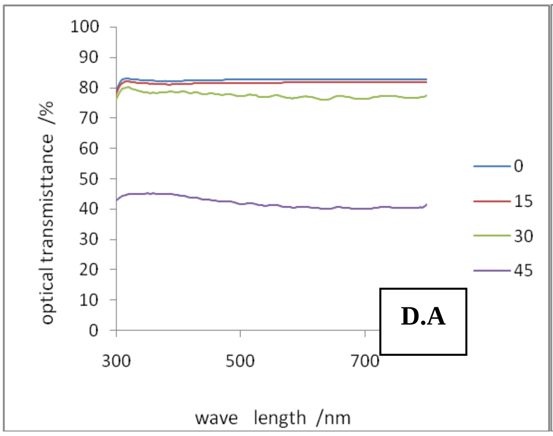
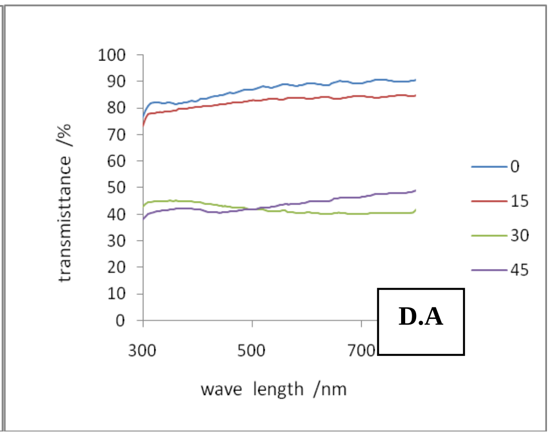
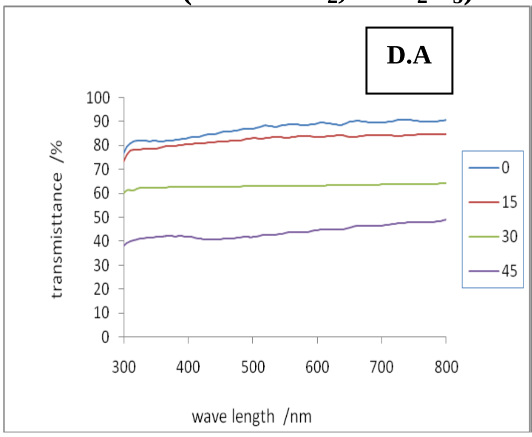


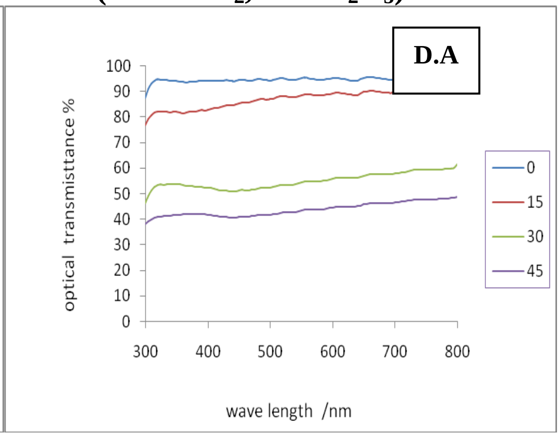
Figure 9. Effect of weathering condition on contact angle for (50% TiO_2,50%V_2O_5) results after exposure to 4 months weather condition, and deposition angle.



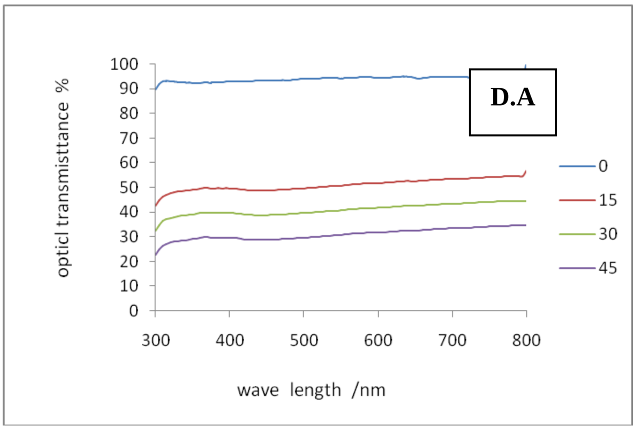


a:(100%TiO_2,0%V_2O_5) **b:(75% TiO_2,25 %V_2O_5)**





c:(50% TiO_2, 50%V_2O_5) **d: (25% TiO_2, 75%V_2O_5).**



e: (0 % TiO_2, 100 %V_2O_5)

Figure 10. Effect of wave length and deposition angle on optical transmittance of thin film at different concentration.