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EFFECT OF UV RADIATION DURATION AND MOLECULAR WEIGHT TO HYDROPHOBICITY AND SURFACE ROUGHNESS OF POLYSTYRENE COATING ON QCM SENSOR

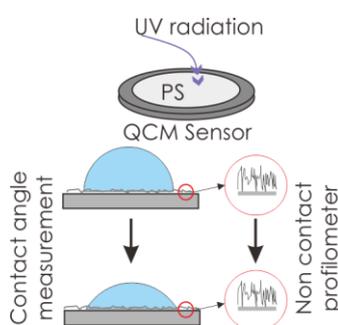Setyawan P. Sakti*, Layli Amaliya, Nike F. Khusnah, Masruroh

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Graphical abstract

Abstract

Hydrophobicity is one of important solid surface properties for the development of Quartz Crystal Microbalance (QCM) biosensor. Hydrophobicity plays a role in the biomolecule immobilisation. Polystyrene is one of the coating materials used in the QCM biosensor, where the sensitive biomolecule material is immobilised. Hydrophobicity and surface roughness can be controlled by many methods. In this work, we investigated the effect of the polymer molecular weight and UV radiation on the surface roughness and hydrophobicity. The polystyrene with a molecular weight of 35,000 g/mol, 192,000 g/mol, and 280.000 g/mol were solved in toluene with a concentration of 3%, 5%, and 7% and coated using spin coating method on top of the QCM sensor. The coated polystyrene on QCM sensor was irradiated using UV lamp with a wavelength of 254 nm. The contact angle of polystyrene before and after UV irradiation was measured using contact angle instrument and the surface roughness is measured using non-contact optical profilometer. The result shows that the higher molecular weight of polystyrene led to more hydrophobic surface. Radiation under UV light increases the hydrophobicity of polystyrene surface. The surface roughness of the polystyrene on top of the sensor is not affected by UV irradiation.

Keywords: Hydrophobicity, polystyrene, molecular weight, UV radiation, contact angle

1.0 INTRODUCTION

Polystyrene is widely used in many applications, including in the sensor development. Polystyrene can be used as itself and also modified with other materials. Polystyrene modification, for example, is used as part of a gas sensor [1]. Polystyrene is also widely in the study of interaction among biomolecule. Study on the biomolecule immobilisation on the polystyrene surface and modified polystyrene surface can be found in many works. Plasma treatment [2, 3], laser ablation [4–7] and UV irradiation [5, 8–10] have been reported to modify polystyrene surface properties to achieve a better biomolecule adsorption on the surface. The developed method can be from a simple system to complex system. Among many

methods, UV radiation is the easiest way to modify the polystyrene surface property.

In the CM biosensor development, immobilisation of the biosensitive material is one of the important factors. The biomolecule immobilisation was done in many different ways. All methods benefit from the sensor surface layer interaction with the biomolecule. The sensor surface, using an additional coating layer or only the metal electrode surface, functions as immobilisation matrix.

Physical adsorption is one from many immobilisation methods. Physical adsorption mechanism can use polystyrene coating as immobilisation matrix [11–13] or with a modification [5, 14–16]. Hydrophobicity of the polystyrene coating on sensor surface plays an important role

not only for biomolecule adsorption [5, 17, 18] but also the sensor performance.

Hydrophobicity is one of the solid properties which can be described by solid surface properties. Hydrophobicity can be measured by the contact angle of water on the solid surface. The contact angle is the angle made by the liquid surface to the solid surface at the point where both liquid and solid surface meet. A surface is called hydrophobic if the contact angle of water on it is greater than 90°. Conversely, if the contact angle of water on it is less than 90°, the surface is hydrophilic.

Polystyrene surface hydrophobicity affected by many factors including preparation process. A study by Huan *et al.* [19] showed that polystyrene layer resulted from different solvents (DMF and THF) result in different contact angled and also surface roughness [20]. It is also known that UV radiation can affect the polystyrene surface hydrophobicity [8] because the ultraviolet radiation could degrade polystyrene [8, 21, 22]. Photodegradation mechanism of polystyrene can be found in many references [21, 23].

There were no information on the role of the polystyrene molecular weight and UV irradiation in air condition to the polystyrene hydrophobicity and surface roughness to be applied for QCM sensor. In this research, the hydrophobicity of QCM sensor with polystyrene coating was investigated by using different polystyrene molecular weight, i.e., 35,000 g/mol, 192,000 g/mol and 280,000 g/mol, and by irradiated with 254 nm UV light for one hour and two hours. Short time irradiation was done to avoid severe damage to the polystyrene surface. Topography investigation has also been done to know the morphology of polystyrene surface and the relation with its hydrophobicity.

2.0 METHODOLOGY

We used a commercially crystal resonator with a silver electrode. The resonator was purchased from PT Greatmicrotama, Surabaya, Indonesia. Three variations of polystyrene molecular weight, i.e., 35,000 g/mol, 192,000 g/mol and 280,000 g/mol for coating film on QCM are used. Those molecular weight represents a low molecular weight to high molecular weight of polystyrene. The polystyrene was purchased from Sigma-Aldrich. Toluene was used as polystyrene solvent.

The equipment used were micropipette, oven, ultrasonic cleaner, spin coater, 39 Watt UV lamp (Sterilight S36RL) with a peak wavelength at 245nm, non-contact topography measurement system (Polytec TMS-1200 TopMap µ.Lab), and Contact Angle Measurement System. The UV wavelength of 245 nm is in wavelength spectrum of the polystyrene absorption of 230nm to 280 nm.

Polystyrene was dissolved in toluene solvent at a concentration of 3%, 5%, and 7% mass polystyrene. Our preliminary experiment shows that lower concentration results in a thinner polystyrene layer which results in a condition where the polystyrene does not well cover the sensor surface. Higher concentration of polystyrene results in an inhomogeneous surface thickness. To advance dissolve rate, we used an ultrasonic cleaner for about 15 minutes. Polystyrene solutions subsequently deposited on QCM surface using spin coater at a rotation speed of 3000rpm. The QCM sensor with polystyrene coating is heated in the oven to evaporate any solvent residue on top of the coating layer.

Hydrophobicity alteration is done by irradiating the QCMs with polystyrene coating under UV lamp for 1 and 2 hours with a distance of 2 cm. Irradiation is done in air. Before and after the irradiation, the contact angle of the polystyrene surface is measured using contact angle measurement system based on goniometry.

The contact angle measurement was done using the contact angle system in the following procedure. At first, the QCM sensor was placed on the holder located on the stage of contact angle instrument. Through the optical lens connected to a digital video capturing system, the alignment of the sensor surface to the capturing camera was observed. The position of the sensor was arranged so that the surface of the sensor is perpendicular to the camera.

After the optimally horizontal alignment had been achieved, a 30 µL of distilled water was dispensed on the QCM surface using micropipette. The water droplet is illuminated using visible light to enhance the visibility and contrast of the image,
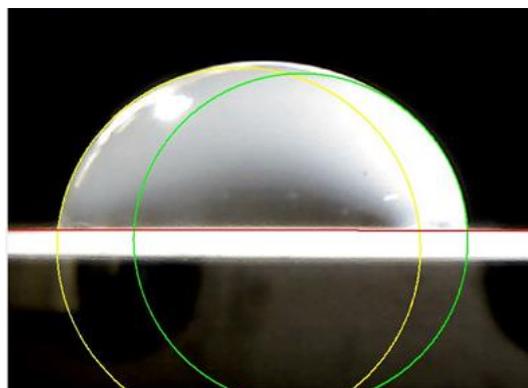

Figure 1 Droplet image and contact angle measurement result suing circle fit model. Left angle 84.394° and right angle 86.914°

Figure 1 shows a sample of droplet image and measured contact angle. Due to the wide droplet sample and surface flatness, the measured contact angles of the left side and right side of the droplet are not always the same. Alignment is done to get the best surface alignment and it shows that the difference between the measured contact angle of the left side and the right side of the droplet is close to zero.

To investigate the morphology of polystyrene surface and contribution of surface roughness to polystyrene hydrophobicity, we used a non-contact Topography Measurement System. The Polytec TMS-1200 TopMap µ.Lab is a non-contact

optical interferometry system with 10x magnification.

3.0 RESULTS AND DISCUSSION

Statistical analysis was used to evaluate the contact angle among different samples. Table 1 shows the resulted contact angle measurement for each polystyrene with different concentration, molecular weight, and UV irradiation. As there is no replication of the experiment, analysis of variance with two non-replicated factors was used. The analysis is conducted using Microsoft Excel.

Table 1 Average surface contact angle from different molecular weight and UV irradiation (A: 35,000 g/mol , B: 192,000 g/mol , C : 280,000 g/mol)

Concen-tration	MW	No UV	UV (1 hour)	UV (2 hours)
3%	A	85.4±0.8	84.1±0.40	65.7±0.83
	B	86.3±0.35	74.6±0.74	66.7±1.0
	C	91.5±0.98	82.6±0.64	70.1±0.69
5%	A	82.4±1.4	81.7±0.24	68.8±0.50
	B	86.2±0.59	73.5±0.33	64.8±0.89
	C	91.9±0.99	77.7±1.0	73.2±1.3
7%	A	86.0±0.93	83.6±0.52	67.8±1.2
	B	87.2±0.77	76.5±0.47	67.0±0.84
	C	92.6±1.5	80.8±1.1	73.8±1.8

Table 2 Analysis of Variance of surface contacts angle of polystyrene surfaces without UV irradiation (MW: molecular weight)

Source of Variation	SS	df	MS	F	P-value	F crit
Concen-tration	4.560	2.000	2.280	2.314	0.215	6.944
MW	88.057	2.000	44.028	44.691	0.002	6.944
Error	3.941	4.000	0.985			
Total	96.558	8				

Note: SS: sum of squares; df: degree of freedom; MS:mean square; F: F-statistic value; P-value:probability value; F crit: critical value of statistical test at 0.05 significance level

Table 2 shows the Analysis of Variance (ANOVA) with two-factor without replication for the contact angle of the polystyrene surface without UV irradiation, the analysis results in no significant different in contact angle between the sample with difference concentration (the analysis gives no significance result in contact angle between the sample with different concertation) (ANOVA P-value = 0.215). It means that variation of concentration does not affect the surface contact angle. UV irradiation in this experiment only affects the surface property of the polystyrene not the bulk property of the polystyrene. The weak van der Walls and electrostatic force between the surface and water play role in the interaction between material surface and water [24]. On the other hand, there is a significant difference in contact angle value

between each difference molecular weight (ANOVA P-value = 0.0018).

It also can be seen in Table 1, the contact angle of polystyrene surface without UV irradiation ("No UV") increases as molecular weight increases. The larger molecular weight results in a larger contact angle. This result means that larger molecular produces a more hydrophobic surface.

Analysis of variance test results for the contact angle of the polystyrene surface after 1 hour and 2 hours UV irradiation are presented in Table 3 and Table 4. For the contact angle of the polystyrene surface after 1-hour irradiation, the significant difference is found in different molecular weights. After 2 hours irradiation, it can be seen that the contact angle is also affected by the molecular weight.

Based on those results, it can be seen that selection of the molecular weight is important in an attempt to achieve different hydrophobicity of the polystyrene surface. Different coating thickness does not affect the surface contact angle, whether the surface is radiated or not radiated with UV.

Table 3 Analysis of Variance of surface contact angle of polystyrene surface with 1 hour UV irradiation (MW: molecular weight)

Source of Variation	SS	df	MS	F	P-value	F crit
Concen-tration	15.171	2.000	7.586	5.623	0.069	6.944
MW	107.163	2.000	53.581	39.717	0.002	6.944
Error	5.396	4.000	1.349			
Total	127.730	8.000				

Table 4 Analysis of Variance of surface contact angle of polystyrene surface with 2 hours UV irradiation (MW: molecular weight)

Source of Variation	SS	df	MS	F	P-value	F crit
Concen-tration	6.711	2.000	3.356	1.463	0.334	6.944
MW	64.209	2.000	32.104	13.992	0.016	6.944
Error	9.178	4.000	2.294			
Total	80.098	8.000				

The effect of the radiation on the polystyrene contact angle which indicates its hydrophobicity is analysed using the Analysis of Variance. The contact angle variation caused by UV irradiation for 1 hour and 2 hours, and also without irradiation were used. As the polystyrene concentration does not affect the surface contact angle, the analysis will be done using a single factor ANOVA to see the significant difference of the treatment by UV irradiation to the polystyrene contact angle.

Table 5 shows the P-value of the data calculated using single factor analysis of variance. For all the polystyrene used in this experiment, the P-value of the data shows a value of close to zero. This result indicates that there is a significant difference in the polystyrene contact angle caused by UV radiation.

The different exposure time of UV radiation results in different surface contact angle of the polystyrene coating.

In the Table 1 column "UV (1 hour)", we can see that after polystyrene surface was irradiated by UV for one hour, the contact angles were decreased as a result of the carbonyl/carboxyl species caused by UV irradiation [8]. Polystyrene with larger molecular weight has a larger decrease of contact angle after irradiated by UV for 1 hour and 2 hours. Higher molecular weight means longer polymer chains. Therefore, when the polymer with longer chains irradiated by UV, the UV radiation breaks more chains compare with the shorter molecule chain.

Table 5 Resume of Analysis of Variance of surface contact angle of polystyrene surface with different molecular weight at different UV irradiation duration (No radiation, 1-hour irradiation, and 2 hours irradiation)

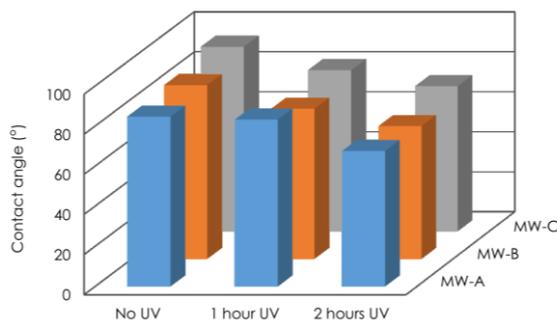| Molecular Weight	Statistical value		
	F	P-value	F crit
35,000 g/mol	104.95	2.146E-05	5.1432
192,000 g/mol	234.16	2.024E-06	5.1432
280,000 g/mol	82.07	4.385E-05	5.1432

Figure 2 Contact angle of polystyrene surface before and after UV irradiation (MW-A: 35,000g/mol, MW-B: 192,000g/mol and MW-C: 280,000 g/mol)

It is known that polystyrene degradation by UV results in carbonyl (C=O) and hydroxyl (O–H) group [21]. Carbonyl and hydroxyl group are polar and like water too. It was also known that carbonyl and hydroxyl are hydrophilic groups. Thus, when the surface was covered with carbonyl and hydroxyl group from polystyrene degradation, the water would spread on the surface and create a smaller contact angle. The reason is that polar molecules effectively bound with polar molecules. The longer polymer chains result in more carbonyl and hydroxyl group on the surface after radiation. Therefore, the polystyrene surface with longer molecule chain tends to be more hydrophilic than the polystyrene with shorter molecule chains.

Longer irradiation time theoretically results in more molecule chain of the polystyrene to be broken. That chain broke, in turn, affected the polystyrene surface to be more hydrophilic. It means the contact angle should be smaller.

Data in Table 1 shows that there is a decrease in the contact angle for all polystyrene after UV irradiation for 2 hours. Figure 2 shows the contacts

angle change of each polystyrene after UV irradiation at different irradiation time. The contact angle of the polystyrene surface after 2 hours irradiation is smaller if compared to the one after 1-hour irradiation.

Macroscopic view of the surface morphology of the coating can be seen in Figure 3. It can be seen that the whole sensor surface is well covered with the polystyrene coating. There is no significant difference in the coating morphology. UV irradiation does not affect the surface of the coating. Optically, there is no significant difference between the coating layer before and after UV irradiation.
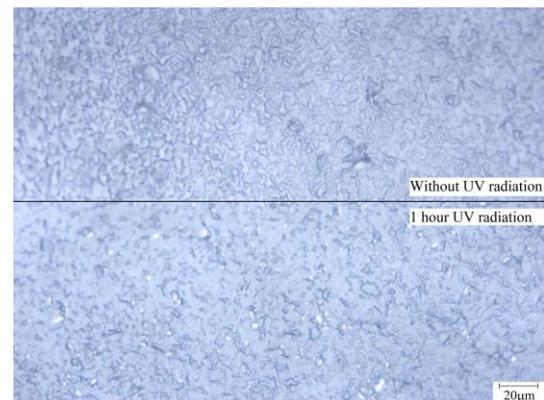

Figure 3 Image of the polystyrene sensor coating with 50× magnification

According to Wenzel [25], the hydrophobicity of solid surface can be affected by surface roughness. For hydrophobic material, rougher surface results in the more hydrophobic surface. To investigate the roughness effect of polystyrene surface to its hydrophobicity, we used TMS-1200 TopMap μ.Lab which gives roughness data of polystyrene with various molecular weight.

Figure 4 shows an example of the measured surface roughness using TMS with 10x objective magnification. The scanning area is 900 μm x 700 μm. It can be seen that the sensor surface in the area is well covered with the polystyrene layer with high homogeneity. There is no area with step height coating thickness. Measurement of the electrical impedance of the sensor before and after coating results in no impedance difference. It means that the surface coating is well distributed on top of the sensor. Therefore, surface roughness measurement can be done in any place of the targeted area. The presentation of the surface roughness data is cropped at 100 μm horizontal length to clarify the surface roughness.
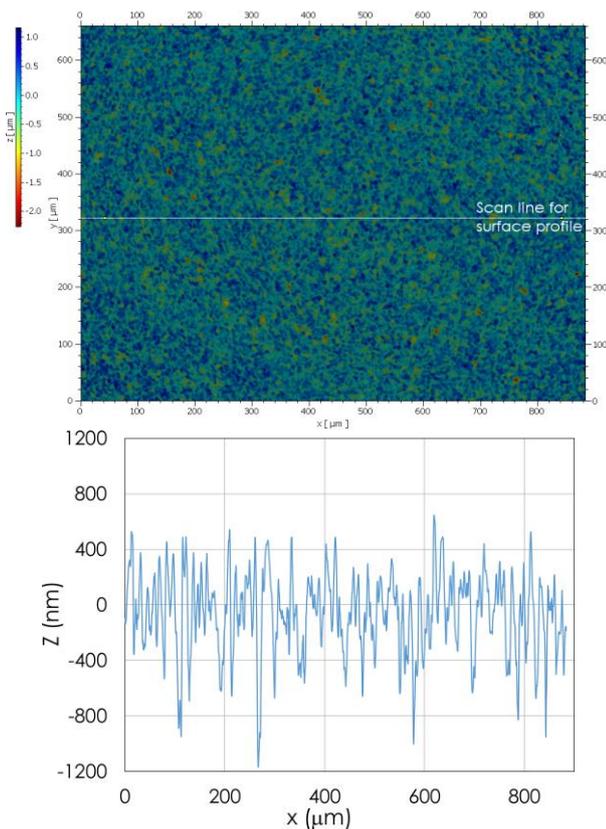
Figure 4 The surface profile of sensor surface made from polystyrene with 35,000 g/mol without UV irradiation taken using TMS 1200 TopMap μ.Lab

The surface profile of the samples is presented in Figure 5 to Figure 7 for different polystyrene molecular weight and UV irradiation duration. Note that the surface profile line not taken from the same position. From Figure 5 to Figure 7, it can be seen that the surface profiles have a similar pattern. There is a random variation in the surface depth. The surface profile shows a smooth change in the vertical direction, where the change of the surface height is less than 1 μm.

The surface roughness of the polystyrene surface measured using the TMS 1200 is presented in Figure 8. It can be seen from Figure 8 that there is no significant difference of the surface roughness to the molecular weight. The difference in surface roughness from molecular weight from 35,000g/mol to 280,000g/mol is still under their standard deviation. P-values among molecular weight variation on each treatment are higher than 0.05.
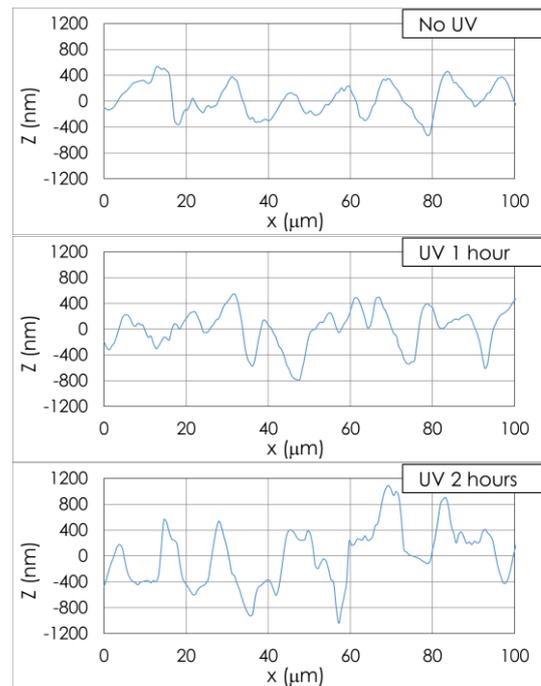

Figure 5 The surface profile of sensor surface made from polystyrene with 35,000 g/mol molecular weight before and after UV irradiation
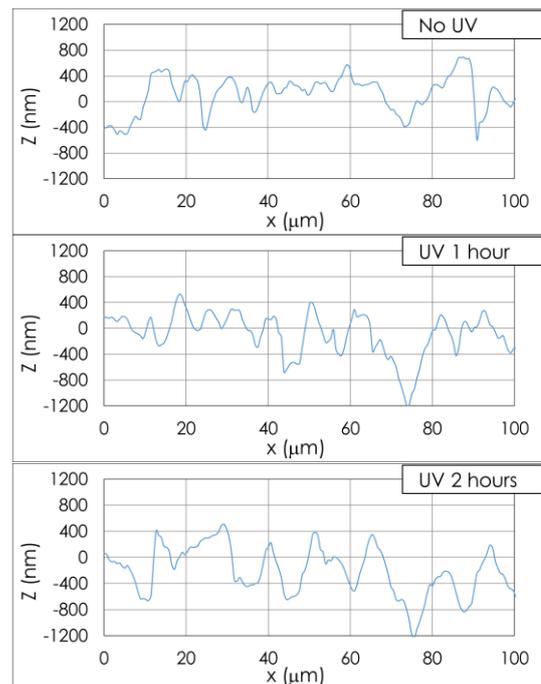

Figure 6 The surface profile of sensor surface made from polystyrene with 192,000 g/mol molecular weight before and after UV irradiation
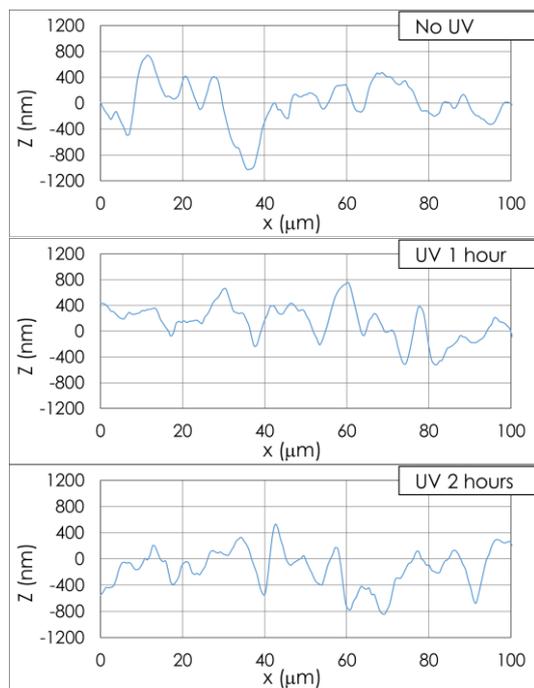
Figure 7 The surface profile of sensor surface made from polystyrene with 280,000 g/mol molecular weight before and after UV irradiation
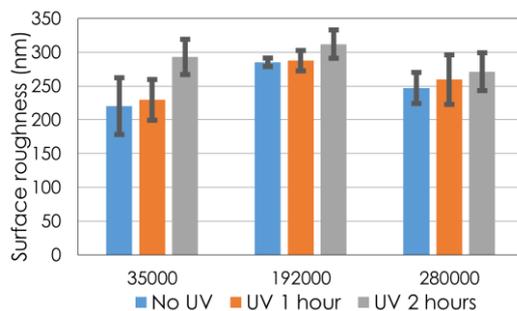

Figure 8 Surface roughness and its standard deviation (black line) before and after UV irradiation for 1 hour and 2 hours

Comparing with the result of the contact angle measurement, we can say that the hydrophobicity is not only affected by the surface morphology but also affected by other non-geometrical interaction between the surface and the water. It seems that the ionic force of the surface is responsible for attracting or repelling water molecule plays more important role in the surface contact angle in our case.

Statistical analysis is done to evaluate the significance of the surface roughness change caused by UV irradiation for 1 hours and 2 hours. The statistical value using analysis of variance for the measured data is presented in Table 6. From the Table 6, it can be seen that all of the P-value of the statistical test results are higher than 0.05. It means that at a significant level of 0.05, statistically there is no significant difference in the surface roughness of the polystyrene coating after 1 hours and 2 hours UV irradiation.

Table 6 Analysis of variance of the polystyrene surface after UV irradiation for 1 hour and 2 hours (MW-A: 35,000g/mol, MW-B: 192,000g/mol and MW-C: 280,000 g/mol

Source of Variation	SS	df	MS	F	P-value	F crit
MW-A	9406.52	2.000	4703.26	4.072	0.055	4.256
MW-B	3900.56	2.000	1950.28	2.280	0.158	4.256
MW-C	272.42	2.000	136.21	0.209	0.815	4.256

This result is important as surface roughness change may also introduce different effects to the QCM performance in contact with a liquid. The statistical analysis also tells that the UV irradiation to higher molecular weight has less effect on the surface roughness. One can modify the polystyrene contact angle without a significant change in the surface roughness by controlling the length of UV radiation. In the case of Polystyrene under UV irradiation, the surface contact angle change is dominated by the molecule degradation of the polymer compare to the surface roughness. This result implies that one can control the surface contact angle of polystyrene coating layer for the QCM sensor without affecting the surface roughness.

4.0 CONCLUSION

Hydrophobicity of the polystyrene surface on QCM sensor is affected by the molecular weight of the polystyrene. Higher molecular weight of the polystyrene results in a higher contact angle. UV irradiation on the polystyrene makes the surface becoming more hydrophilic compared to the surface without UV irradiation. Longer UV irradiation time results in lower surface contact angle of the polystyrene. UV irradiation up to 2 hours increases the surface roughness of the polystyrene surface but statistically not significant.

Acknowledgement

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