International Journal of Research in Advanced Engineering and Technology
ISSN: 2455-0876; Impact Factor: RJIF 5.44
Received: 02-01-2021; Accepted: 21-01-2021; Published: 25-02-2021
www.newengineeringjournal.in
Volume 7; Issue 1; 2021; Page No. 17-22

Detecting the influence of polymeric additives on surface free energy of aged asphalt cement

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Abstract

Physicochemical surface characteristics of asphalt binder material can be well represented by surface free energy (SFE) which is a useful tool for the selection of the moisture-resistant constituent material. Implementation of polymeric additives can reserve or even improve the quality of aged asphalt binder. In the present investigation, an attempt has been made to implement polymeric additives such as crumb rubber (CR) and Low-Density Polyethylene (LDP) for recycling the aged binder of 40-50 penetration grade. The influence of additives was assessed with the aid of surface free energy determination. Sessile drop and Wilhelmy plate techniques have been implemented. It was observed that both additives exhibit higher contact angle and surface free energy when sessile drop technique is implemented regardless of the additive content. However, the contact angle decreases after implementation of additives when Wilhelmy plate technique was used. The SFE increases by (110, 392, and 431) % and (6.9, 40.7, and 66) with the addition of (0.5, 1.0, and 1.5) % of polyethylene when Sessile drop and Wilhelmly plate methods was used respectively. The SFE increases by (243.6, 351.3, and 131.2) % and decreases by (6.7, 18.1, and 51) with the addition of (0.5, 1.0, and 1.5) % of crumb rubber when Sessile drop and Wilhelmly plate methods was used respectively.

Keywords: polymeric additives; surface free energy; asphalt cement; sessile drop, wilhelmy plate

Introduction

According to Wasiuddin *et al.*, 2007 [1], SFE is defined as the amount of external work done on a material to create a new unit surface area in a vacuum or the energy associated with the intermolecular forces at the interface between two media or it quantifies the disruption of intermolecular bonds that occur when a surface is created. To achieve a proper type of adhesion and resistance to moisture damage, variations in the chemical nature of binders by implementation of additives is required as stated by Ahmad, 2011[2]. High silicon oxide content aggregates, quartz and granite origin makes the aggregates hydrophilic which are difficult to coat with asphalt binder than basic hydrophobic aggregates such as limestone as reported by Hofko *et al.*, 2019 [3]. Generally, the dispersive component for binders varies from 12 to 35 ergs/cm^2 and for aggregates from 35 to 80 ergs/cm^2 as addressed by Handle *et al.*, 2017 [4]. Base components are higher than acid components for aggregates; for most binders, acid components are higher than base components. This difference between the aggregate and binder components contributes to the adhesion bond energy between the aggregate and binder The SFE components of the aggregates and asphalt binder can be measured using different techniques as stated by Little and Bhasin, 2006 [5]. Among others, these include the Wilhelmy plate, and sessile drop techniques. The results acquired from these tests are further utilized to approximate the bond energy of adhesion between the aggregate and asphalt binder with and without the presence of moisture. The total surface free energy of a material is divided into three components. Based on the type of molecular forces acting on the surface, these three components include the Lewis acid component, Υ^{+}, the Lewis base component, $\Upsilon-$; and the nonpolar component which is also referred to as the Lifshitz–van der Waals (LW) component, Υ^{LW}. The total SFE of the material, Υ total, is calculated using the following equation according to Kakar *et al.*, 2019. [6]:

$$\Upsilon \text{ total} = \Upsilon^{LW} + 2\,(\Upsilon^{+}\,\Upsilon^{-})^{0.5} \qquad \text{----- (1)}$$

The most popular, widely used, and result-oriented method for the evaluation of asphalt binder surface free energy is the measurement of contact angles for both testing techniques using different probe liquids. Li *et al.*, 2019 [7] stated that the contact angle refers to the tangent of the liquid interface at the intersection of the liquid, and solid phases. The angle between the tangent and the solid–liquid boundary of the liquid is a measure of the degree of wetting. If the contact angle is lower than 90°, the solid surface is hydrophilic, that is, the liquid can easily wet the solid, and the smaller the angle, the better the wettability. If the contact angle is more than 90°, the solid surface is hydrophobic, that is, the liquid does not easily wet the solid and easily moves on the surface. Zhang and Luo, 2019 [8] reported that in the SFE theory, the asphalt mixture resistance against the loss of asphalt cement cohesion and asphalt cement-aggregate adhesion is measured in wet and dry conditions; this strength of materials is naturally dependent on the basic characteristics of the materials. Hossain *et al.*, 2019 [9] studied the impacts of aging and rejuvenation on fundamental properties of asphalt binders. A Performance Grade 64-22 binder was aged to simulate the aging of asphalt mixes during construction and in-service conditions. The contact angles between binder and three probe liquids, was measured using a sessile drop device. This property was then used to estimate surface free energy components of the control binder, aged binders, and rejuvenated binders. The data showed that as asphalt ages, the contact angle between (distilled water) and the asphalt surface increases; however, for the other two probe liquids (formamide and glycerol), no pattern was observed. For the surface free energy components of the control binder, the aged binders, and the binders with rejuvenators, the contribution from Lifshitz–van der Waals components were much higher than those from acidic and basic components. Cheng *et al.*, 2002 [10] investigated the concepts of SFE measurement and its application in asphalt mixtures. The results obtained from the study indicated that thermodynamic changes in SFE of adhesion and cohesion are directly related to de-bonding at the asphalt cement-aggregate interface and crack occurrence in mastic. Another study by Aguiar-Moya *et al.*, 2019 [11] on a PG 64-22 binder modified with several additives including polymers, nanomaterials, and adhesion promoters addressed that testing was performed on binder after rolling thin-film oven (RTFO) aging. The surface free energy of the asphalt and the aggregate, with and without the presence of water, was estimated also. The results indicated an increase in strength of adhesion associated with the aging process. Ageing can also be the positive reason for an improved ability to resist distress, like permanent deformation as addressed by Little *et al.*, 2018 [12] because of the increase in stiffness. Therefore, many authors, Sarsam, 2007 [13] and Alisov *et al.*, 2020 [14] consider better knowledge on ageing behavior and ageing susceptibility of asphalt binders of utmost importance for designing more durable asphalt pavements. It was found by Koyun *et al.*, 2020 [15] that after ageing, equivalent modulus temperature increases, and the corresponding phase angle decreases. This means that ageing increases stiffness and elasticity. The test results indicate that rheological and binder properties follow an approximately linear trend for increasing ageing condition of the material. Interestingly, the highest impact of oxidation happens during the first ageing step, whereas all subsequent ageing conditions have approximately similar but reduced impact. Yang *et al.*, 2014 [16] explored the aging mechanism of bitumen and stated that the aging of bitumen comes from three aspects: Loss of volatiles, dehydrogenation, and oxidation. It was concluded that due to chemical variation, aged bitumen has higher modulus and lower phase angle, showing a solid-like rheological behavior, which is observed by increasing viscosity and brittleness as well as decreasing flexibility. Molenaar *et al.*, 2010 [17] studied the effects of well-known short-term and long-term laboratory aging procedures on the rheological characteristics and chemical composition of binders. Test results are compared with the rheological characteristics and chemical composition of field aged binders. The results obtained on field aged binders are also compared with the results of laboratory aging protocols in which a special weather meter was used. The results clearly show that none of the laboratory procedures is capable of simulating long-term field aging. At best two years field aging could be simulated. Mansourian and Gholamzadeh, 2017 [18] used polypropylene nano composite for improving the moisture susceptibility of the asphalt mixtures. The experimental design included one base asphalt binder, two types of aggregates (granite and limestone) and 2% of nano composite. The results of surface free energy test indicated that the nano composite could be used to improve the moisture susceptibility of the asphalt mixtures.

The aim of the present investigation is the assessment of the influence of polymeric additives (polyethylene and crumb rubber) on the surface free energy of the aged asphalt binder using sessile drop and Wilhelmy plate techniques.

Materials and Methods

The materials implemented in this investigation are used in pavement construction.

Asphalt Cement

Asphalt cement binder of penetration grade 40-50 was obtained from Doura oil refinery, south of Baghdad and implemented in the present investigation. The physical properties are demonstrated in Table 1.

Table 1: Physical Properties of Asphalt Cement Grade (40-50) as per ASTM, 2015 [19]

Property	Units	Test	Result
Penetration at (25°C,100mg,5sec)	0.1mm	ASTM D5	46
Softening point(Ring & Ball)	°C	ASTM D36	51
Ductility at (25°C,5cm/min)	Cm	ASTM D113	118
Flash Point (cloven open cup)	°C	ASTM D92	286
Specific Gravity at (25°C)	…	ASTM D70	1.042
After Thin-Film Oven Test ASTM D- 1754			
Penetration of Residue.	0.01mm	ASTM D-5	31
Ductility of Residue	Cm	ASTMD-113	93
Loss in Weight (163 °C, 50g, 5h).	%	…	0.175

Low-Density Polyethylene (LDP)

A Polymer produced by SABIC was recommended by the literatures to be suitable for blending with asphalt. Table 2 exhibit the Mechanical properties of (LDP) while Table 3 show the thermal properties of Polyethylene (Low Density).

Table 2: Mechanical properties of Low-Density Polyethylene

Properties	Unit	Value
Tensile strength	MPa	10
Tensile Elongation	%	>350
Flexural Modulus	MPa	8
Hardness (Shore D)	....	50

Table 3: Thermal properties of Low-Density Polyethylene

Properties	Unit	Value
Melting Temperature	°C	<180
Storage Temperature	°C	160
Vicat Softening point	°C	88
Brittleness Temperature	°C	<-175

Crumb Rubber (CR)

The crumb rubber was produced by mechanical shredding at ambient temperature. It was obtained from tires factory at AL-Najaf governorate. This type of rubber is recycled from used tires. Table 4 show the grain sizes distribution of crumb rubber.

Table 4: Grain Sizes Distribution of Crumb Rubber

Sieve size (mm)	1.18	0.6	0.3	0.075
% passing by weight	100	78	25	Zero

Probe Liquids

Three probe liquids were used in this study, De-ionized water, Formamide, and Glycerol. All probe liquids were with known surface energy properties and fulfill the requirements for surface energy testing. Chemical properties of the prob liquids are shown in the Table 5. The selected probe liquids were used by several researchers such as Liu *et al.*, 2017 [20]; Zhang and Luo, 2019 [8].

Table 5: Probe Liquids Properties as provided by the supplier

Properties	Deionized water	Glycerol	Formamide
Chemical formula	H_2O	$C_3H_8O_3$	CH_3NO
Molar mass	18.015 g·mol^{-1}	92.09 g·mol^{-1}	45.04
Appearance	clear, colorless	colorless liquid hygroscopic	clear,colorless
Odor	Odorless	Odorless	Odorless
Density	0.9950 g/ cm^3 at 25 °C	1.261 g/cm^3	1.134 g/mL at 25 °C (lit.)
Melting point	0°C (32° F)	17.8 °C (64.0 °F; 290.9 K)	2-3°C
Boiling point	100°C, 760 mm Hg	290 °C (554 °F; 563 K)	210 °C
Solubility in water	Completely miscible	miscible	Miscible
Vapor pressure	23.756 mmHg (25°C)	0.003 mm Hg (50°C)	0.08 mm Hg (20 °C)
Viscosity	0.8976 Pa·s	1.412 Pa·s	3.76m.Pa.s

Aged and Modified Asphalt Cement Preparation

The asphalt cement of penetration grade 40-50 was subjected to thin film oven test. The thin film oven test as per ASTM D 1754, 2015 [19] is implemented to evaluate the changes in physical properties of asphalt cement when its exposure to short-term aging process (heat and air) during conventional hot mixing in asphalt plants. A set of 50-gm of binder samples resulting in a thin film thickness of 3.2 mm of asphalt cement was placed in a flat container with (140 mm in diameter). The containers were placed on the rotating shelf oven for 5 hours at 163 °C. Polymeric additives (polyethylene and crumb rubber) were added to the aged binder and mixed thoroughly using a high accuracy drill driver with a specified mixing at a blending speed of about 1500 rpm and constant mixing temperature of 150 °C for 60 minutes to promote the chemical bonding of the components. During the blending process, swelling of the asphalt binder could be noticed indicating chemical reaction process going on with the asphalt. The glass slides of (25.4mm×76.2mm×1mm) dimensions were coated with asphalt binder or modified asphalt. Fig. 1 exhibit part of the prepared slides for contact angle measurement.

Fig 1: Part of the prepared slides for contact Angle measurement

Determination of Surface Free Energy Using Sessile Drop Method

A probe liquid is dispensed over a smooth horizontal surface coated with asphalt binder. The image of the drop of liquid formed over the surface of the binder is captured by using a digital camera. Contact angles of the polar and non-polar parts of the asphalt cement are obtained by analyzing the image using image processing software (Surftens 47) software. A static Contact angles measured with different probe liquids are used with equations of work of adhesion to determine the three surface energy components of the asphalt binder as per Little and Bhasin, 2006 [5] procedure.

Determination of Surface Free Energy Using Wilhelmy Plate Method

This method is used to measure dynamic contact angles of the asphalt binder with various probe liquids and to determine surface energy components of the binder. A glass slide (25.4 mm x 76.2mm x 1mm) coated with the asphalt binder and suspended from a microbalance is immersed in a probe liquid. From simple force equilibrium conditions, the contact angle of the probe liquid with the surface of the asphalt binder can be determined. Contact angles are obtained by analyzing the image using image processing software (Surftens 47) software. The test was conducted following the procedure by Hefer *et al.*, 2006 [21].

Results and Discussions

Influence of Ageing on Contact Angles and Surface Free Energy of Asphalt Binder

Table 6 demonstrate the influence of ageing on static and dynamic contact angles of the binder when implementing Sessile drop and Wilhelmy plate methods using various prob liquids. It can be noted that the static contact angle increases after ageing process by (68.7, 151.7, and 64.6) % for (water, acid, and base) prob liquids respectively for Sessile drop method. On the other hand, when implementing the Wilhelmy plate method, the dynamic contact angle also increases after ageing process by (24.2, 8.4, and 3.8) % for (water, acid, and base) prob liquids respectively. It can be observed that the static contact angles after ageing are about twofold higher than the dynamic contact angles regardless of the prob liquids implemented. The increment of contact angle indicates the proper wetting adhesiveness which decline the surface free energy. Such finding agrees with the work by Sarsam and Al-Sadik, 2014 [22]. The control binder is mainly basic and changed to acidic after ageing process, this could be due to increment of asphaltene content.

Table 6: Influence of Ageing on Contact Angles

Testing Method	Static Contact Angle (Sessile Drop)			Dynamic Contact Angle (Wilhelmy plate)		
Prob Liquid	Water	Acid	Base	Water	Acid	Base
Control Binder	63	45	65.7	41.7	55.8	54.4
Aged Binder	106.5	113.3	108.2	51.8	60.5	56.5

Table 7 exhibit the surface free energy data of control and aged asphalt binder calculated for both testing methods. The surface free energy of asphalt binder decreases by (76 and 7.8) % after practicing ageing when tested by Sessile Drop and Wilhelmy plate methods respectively. This may be attributed to the fact that ageing process conducted at (TFOT) had lowered the flexibility of the binder through the loss of volatiles. This can lower the work of cohesion of asphalt binder, and the fracture resistance of the binder was reduced, leaving the binder to be more susceptible to stiffening and cracking. However, the surface free energy of aged binder is lower by 42.5 % when tested by sessile drop method as compared to that tested by Wilhelmy plate method.

Table 7: Influence of Ageing on Surface Free Energy of Asphalt Binder

Testing Method	Surface Free Energy (erg/cm^2)	
	Sessile Drop	**Wilhelmy plate**
Control Binder	16.9	7.7
Aged Binder	4.03	7.01

Influence of Polymeric Additives on Contact Angles of Aged Asphalt Binder

Fig. 2 exhibit the influence of polymeric additives on the static contact angles of aged asphalt binder when sessile drop technique was implemented. It can be noted that the contact angles increased after implementation of additives regardless of prob liquid type and additive type or content. Polyethylene of (0.5, 1.0, and 1.5) % content increases the contact angle regardless of the prob liquid implemented. The contact angles increase by (71.7, 199.5, and 212.2) %, (136, 147, and 148) %, (68.6, 73.8, and 82.2) % for Water, acid and base prob liquids respectively. However, when (0.5, 1.0, and 1.5) % of crumb rubber were implemented, the contact angles increased by (76.3, 71.1, and 91.7) %, (145.5, 173.3, and 154.8) %, (77, 73.5, and 74) % for water, acid and base prob liquids respectively. It can be observed that implementation of crumb rubber exhibit lower contact angle as compared to polyethylene additive which indicate better wettability, more surface free energy, and more resistance to moisture damage.

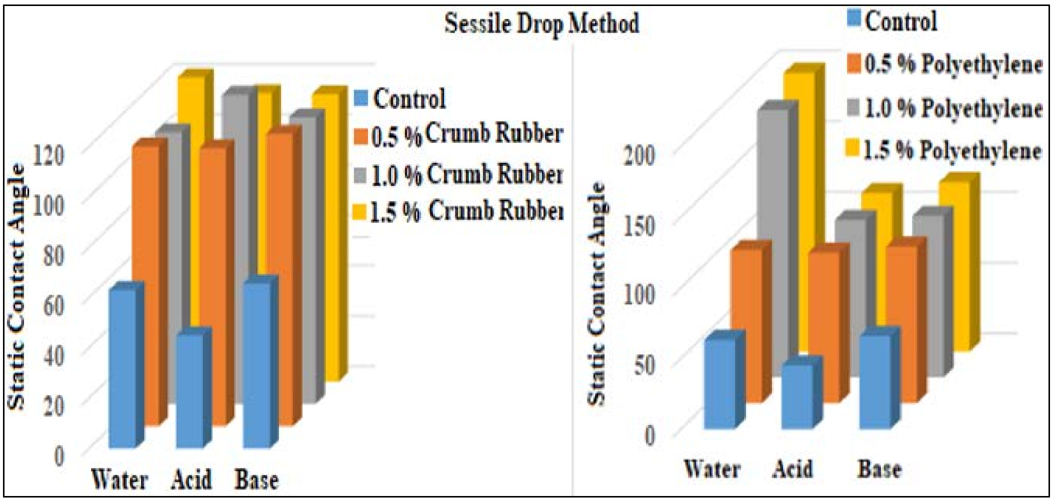

Fig 2: Influence of Polymeric Additives on Contact Angle of Asphalt Cement Using Sessile Drop Method

Fig. 3 exhibit the influence of polymeric additives on the dynamic contact angles of aged asphalt binder when Wilhelmy plate technique was implemented. It can be noted that the contact angles almost decreased after implementation of additives regardless of prob liquid type and additive type or content. Polyethylene of (0.5, 1.0, and 1.5) % content decreases the contact angles regardless of the prob liquids implemented. The contact angles under Wilhelmy plate method decreases the contact angles by (7.2, 13.1, and 7.1) %, (33.1, 36, and 40.8) %, (42, 46.8, and 46.8) % for Water, acid and base prob liquids respectively. However, when (0.5, 1.0, and 1.5) % of crumb rubber were implemented, the contact angles decreased by (23.2 %, 9.6, and 14.3) %, (24.3, 23.4, and 7.8) %, (22.4, 16.3, and 26.8) % for water, acid and base prob liquids respectively. It can be observed that the acid and base component elements of surface free energy are greater than that of deionized water when crumb rubber was implemented. However, deionized water component element of surface free energy is higher than that of acid and base component elements when polyethylene additive was implemented. Such behavior agrees well with the work reported by Li *et al.*, 2019 [7].

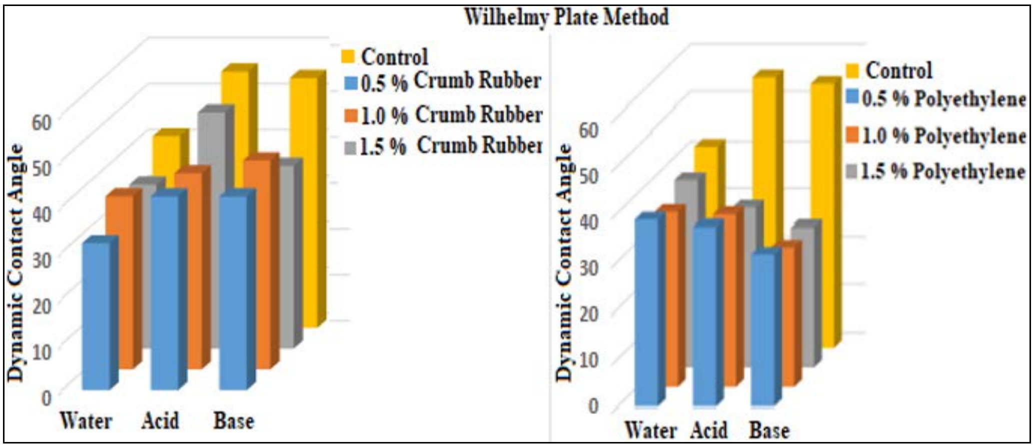

Fig 3: Influence of Polymeric Additives on Contact Angle of Asphalt binder Using Wilhelmy Plate Method

Influence of Polymeric Additives on Surface Free Energy of Aged Asphalt Binder

Fig. 4 exhibit the influence of polymeric additives on surface free energy of asphalt cement. It can be noted that the control binder exhibits higher surface free energy when tested using the Wilhelmy plate technique as compared to that when tested using sessile drop technique. Implementation of polyethylene increases the SFE regardless of the testing method or the additive content. The SFE increases by (110, 392, and 431) % with the addition of (0.5, 1.0, and 1.5) % of polyethylene respectively when Sessile drop method was used. However, when implementing the Wilhelmy plate method, the surface free energy increases by (6.9, 40.7, and 66) % with the addition of (0.5, 1.0, and 1.5) % of polyethylene respectively. It can be observed that polyethylene additive exhibit higher surface free energy when tested under sessile drop than that when tested under Wilhelmy plate method. On the other hand, implementation of crumb rubber exhibit higher SFE than control binder when tested under Sessile drop method, the SFE increases by (243.6, 351.3, and 131.2) % with the addition of (0.5, 1.0, and 1.5) % of crumb rubber respectively, while when the Wilhelmy plate method was used, the SFE decreased by (6.7, 18.1, and 51) % with the addition of (0.5, 1.0, and 1.5) % of crumb rubber respectively. Similar findings were reported by Zhang and Luo, 2019 [8].

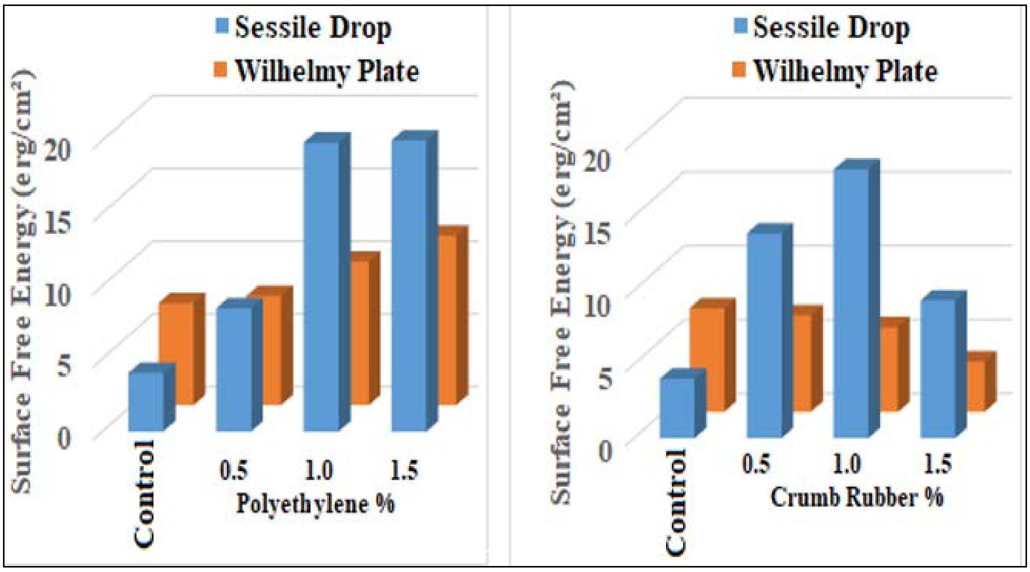

Fig 4: Influence of Polymeric Additives on Surface Free Energy of Asphalt Cement

Conclusions

Based on the testing program and limitations of materials, the following conclusions can be drawn.

1. Sessile drop technique exhibit higher surface free energy than Wilhelmy plate method for control, aged, and polymeric modified asphalt cement.
2. Polyethylene of (0.5, 1.0, and 1.5) % content increases the contact angle regardless of the prob liquid implemented. The contact angles under Sessile drop method increase by (71.7, 199.5, and 212.2) %, (136, 147, and 148) %, (68.6, 73.8, and 82.2) % for Water, acid and base prob liquids respectively. However, when (0.5, 1.0, and 1.5) % of crumb rubber were implemented, the contact angles increased by (76.3, 71.1, and 91.7) %, (145.5, 173.3, and 154.8) %, (77, 73.5, and 74) % for water, acid and base prob liquids respectively.
3. Polyethylene of (0.5, 1.0, and 1.5) % content decreases the contact angles regardless of the prob liquids implemented. The contact angles under Wilhelmy plate method decreases the contact angles by (7.2, 13.1, and 7.1) %, (33.1, 36, and 40.8) %, (42, 46.8, and 46.8) % for Water, acid and base prob liquids respectively. However, when (0.5, 1.0, and 1.5) % of crumb rubber were implemented, the contact angles decreased by (23.2 %, 9.6, and 14.3) %, (24.3, 23.4, and 7.8) %, (22.4, 16.3, and 26.8) % for water, acid and base prob liquids respectively.
4. The SFE increases by (110, 392, and 431) % with the addition of (0.5, 1.0, and 1.5) % of polyethylene respectively when Sessile drop method was used. However, when implementing the Wilhelmy plate method, the surface free energy increases by (6.9, 40.7, and 66) % with the addition of (0.5, 1.0, and 1.5) % of polyethylene respectively.
5. The SFE increases by (243.6, 351.3, and 131.2) % with the addition of (0.5, 1.0, and 1.5) % of crumb rubber respectively when Sessile drop method was used, while when the Wilhelmy plate method was used, the SFE decreased by (6.7, 18.1, and 51) % with the addition of (0.5, 1.0, and 1.5) % of crumb rubber respectively.

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