



Effect of substrate surface roughness on wetting behaviour of vegetable oils

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ABSTRACT

Vegetable oils are mainly used in the heat treating industry due to their environmental friendliness. In the present work the effect of surface roughness on spreading of vegetable oils on stainless steel substrates was investigated. Spreading phenomenon was digitally recorded and analyzed. All of the oils under investigation exhibited power law spreading behaviour of the type: $A = kt^n$, where A , t , k and n represent the drop base contact area, spreading time, constant and exponent, respectively. The coconut and sunflower oils exhibited accelerated kinetics owing to their lower viscosity as compared to palm and mineral oils while peanut oil showed intermediate behaviour. Viscous regime was dominant during spreading of mineral and palm oils as compared to that of coconut oil.

All the oils took longer period of time on rough surfaces than on smooth surfaces to relax to the same degree of wetting. Oils spreading on rough surfaces had to overcome the additional barrier due to asperities of the rough surface. Contact angle decreased with increase in roughness supporting the Wenzel's proposition. The decrease was significant for increase in roughness from 0.25 μm to 0.50 μm for all oils. However, the effect was negligible with further increase in roughness particularly for high viscosity oils. A spread parameter (ψ) is proposed to account for the variation of contact angle with surface roughness of the substrate and momentum diffusivity of the spreading liquid. The result suggested that low viscosity liquids exhibit improved wetting characteristics during spreading on rough surfaces. A model is proposed to estimate dynamic contact angles on substrates having varying surface roughness.

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1. Introduction

A number of industrial processes like lubrication, adhesion, printing, coating, spray quenching, soldering, brazing, etc. essentially involve spreading and wetting processes [1]. Wettability can be defined as the tendency for a liquid to spread on a solid substrate. It can be characterized by the degree and the rate of wetting [2,3]. The degree of wetting indicates the extent up to which the liquid wets the surface and generally quantified in terms of contact angle formed at the three-phase interface. Under equilibrium conditions it is dependent on the surface and interfacial energies involved at the solid/liquid interface. The rate of wetting indicates how fast the liquid spreads on the surface. It is influenced by number of parameters such as surface texture, temperature of the substrate and intrinsic properties of the liquid medium.

The basic mathematical treatment of wetting of a solid surface by a liquid is given by Young–Dupre equation (Eq. (1)) which assumes equilibrium of interfacial energies and also gives an expression for contact angle (θ) formed at the three-phase contact point. Fig. 1 is a schematic sketch of a sessile drop of liquid resting on a

solid surface. A drop of a liquid put on a solid will modify its shape until the equilibrium is attained. The balance of interfacial energies under equilibrium gives

$$\cos \theta = (\gamma_{sv} - \gamma_{sl})/\gamma_{lv} \quad (1)$$

Here γ represents surface energy and subscripts s, l and v indicate solid, liquid and vapour phases, respectively. An angle of 180° indicates zero adhesion between the liquid and surface and therefore represents a total non-wetting condition. For practical purposes, the liquid is said to wet the surface of solid when the contact angle is less than 90°. On the other hand, if the contact angle is greater than 90°, the liquid is considered as non-wetting. In such cases, the liquid drop tends to move about easily on the substrate surface and do not have any tendency to enter into pores or holes by capillary action.

However, the above equation is valid only for an ideal surface which is very smooth and has a well-defined surface tension without taking into the effects like spreading pressure, drop-size, surface roughness, heterogeneity, gas adsorption, etc. More over the total system should be non-reactive (insoluble solid surface and neutral liquid) so that neither physical nor chemical interaction between the solid substrate and spreading liquid will occur [4–7].

The real surfaces are generally rough and behave in a different manner. Within a measured unit surface area on a rough substrate

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Nomenclature

A	base area	γ_{sl}	solid–liquid interfacial energy
k	constant	γ_{sv}	solid–vapour interfacial energy
n	exponent	μ	absolute viscosity
r	average roughness factor	ν	kinematic viscosity
R	base radius	ρ	density
R_a	roughness parameter	θ	quasi-contact angle
t	spreading time	θ_i	initial contact angle
t_r	spreading time for the contact angle to evolve from θ_i to θ_r	θ_r	reference contact angle
ϕ	dimensionless contact angle	θ_w	Wenzel contact angle
γ_{lv}	liquid–vapour interfacial energy	ψ	spread parameter

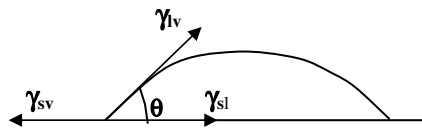


Fig. 1. A sketch showing contact angle at the solid–liquid interface.

there is more surface in contact with the spreading liquid and greater intensity for surface energy. The influence of surface roughness was incorporated by Wenzel using average roughness factor r that represents the factor by which roughness increases the contact area according to

$$\cos \theta_w = r \cos \theta, \quad (2)$$

where θ_w is the apparent angle obtained on a rough surface. The value of r in the above equation is always greater than unity for real surfaces and is equal to unity when the surface is ideal one. From the above equation, it can be seen that the effect of increasing roughness is to enhance the wetting/non-wetting properties of the solid–liquid system. In other words, θ_w increases with roughness if θ is greater than 90° and decreases with roughness if θ is smaller than 90° [8–11].

A number of researchers have studied the kinetics of spreading on real surfaces and modeled the spreading behaviour using a power law correlation given by

$$A = kt^n, \quad (3)$$

where A is the spread area, t is the time, k is a constant and n is exponent [1,9–15]. It was observed that the quantity of liquid did not affect the equilibrium contact angle but it significantly affected the kinetics.

Vegetable oils are mainly used in the heat treating industry due to their environmental friendliness. The earlier studies on heat transfer during quenching with vegetable oils [16,17] revealed that quench severities of coconut and sunflower oils were higher as compared to conventional mineral oil quench medium. It is necessary to investigate whether these oils having higher quench severity show better wettability. In the present work the effect of surface roughness on spreading of various vegetable quench oils on stainless steel substrates was investigated. The experiments were carried out with conventional mineral oil quench medium for comparing its wetting characteristics with vegetable oils. The aim is to study the effect of surface roughness on wetting behaviour and spreading kinetics of vegetable oils.

2. Experimental

Commercially available vegetable oils (peanut, coconut, sunflower and palm) were used as test liquids for wetting studies on 1.5 mm thick stainless steel substrates having dimensions 20 mm × 60 mm. The surfaces of stainless steel sub-

strates were prepared using different grades of silicon carbide papers of varying grit sizes (60, 80, 200, 400 and 600 μm) to obtain surface roughness (R_a) of 0.5 μm (± 0.02), 0.75 μm (± 0.02), 1.00 μm (± 0.03) and 2.00 μm (± 0.04). The surface roughness (R_a) was measured using surface roughness tester (Mitutoyo SurfTest-211). ' R_a ' is the arithmetic mean of the absolute value of the profile departures from the centre line within the evaluation length. Disc polishing with 1 μm lavigated alumina was carried out on the stainless steel substrate to attain a surface roughness of 0.25 μm (± 0.03). Surface roughness values reported in the present work are an average of several independent measurements carried out on the substrate. Mineral oil (SN 150 grade) which is generally used in industrial quench heat treatment was also taken for study for comparing its wetting behaviour with vegetable oils. A dynamic contact angle analyzer, FTA 200, was used for capturing and analyzing the spreading process of a liquid on a solid. The equipment has a flexible video system for measuring contact angle, surface and interfacial energies. A droplet of test liquid was dispensed by the nozzle on to the steel substrate and spreading phenomena was recorded at 60 fps. Captured images were analyzed using FTA software to determine the wetting parameters.

Density and viscosity of the test liquids were determined experimentally using a 25 ml specific gravity bottle and Saybolts viscometer, respectively. Table 1 gives the density and viscosity of various oils used in the present investigation.

3. Results and discussion

Fig. 2 shows the spreading of various oils on a smooth (0.25 μm) stainless steel substrate. Contact angle relaxation was sharp during the initial stages and it became gradual as the system approached equilibrium. The oil started spreading rapidly with a relatively high velocity resulting in sharp increase of base radius. However, within a very short period the spreading rate reduced significantly to almost zero indicating the stabilization of contact angle. This is due to the attainment of equilibrium between the various surface forces under action. Further relaxation of contact angle, increase of spread radius and variation in velocity were negligible.

The wetting behaviour of quench liquids can be well explained using the power law as suggested in the literature available on non-reactive wetting systems [1,6,8,9]. The wetting of a liquid on an insoluble, rigid solid is governed by power relation between contact angle or spread area and time. In the present investigation the spreading kinetics is expressed by the power law: $A = kt^n$, where A is the drop base contact area in mm^2 and ' t ' is the spreading time in seconds. ' k ' is a constant and ' n ' is the exponent.

All the liquids investigated exhibited similar behaviour although the extent of spreading was different for different liquids. The values of k and n were in the range of 8–12 and 0.15–0.2, respectively, for most of the experiments carried out. The magnitude of n for smoother substrate surfaces was found to be around 0.15. On the

Table 1
Density and viscosity of oils at 30 °C

Oil	Mineral	Peanut	Coconut	Sunflower	Palm
Density (kg/m^3)	900	910	910	890	880
Viscosity (stokes)	0.89	0.68	0.40	0.45	0.88

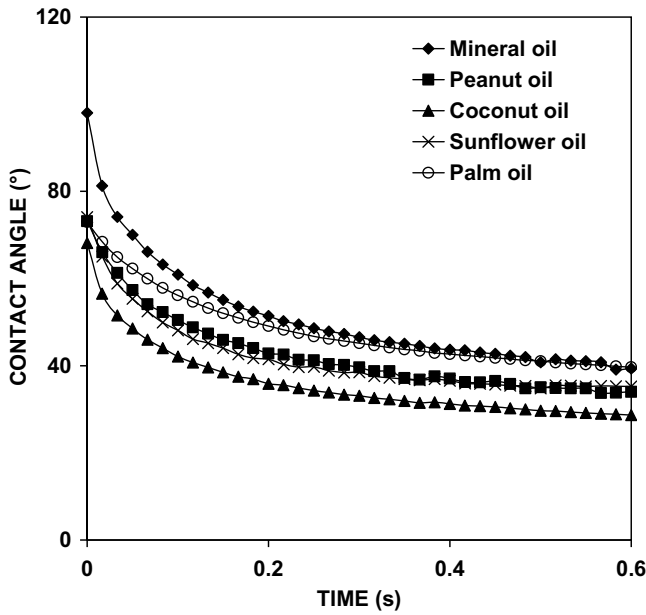


Fig. 2. General relaxation behaviour of various oils on stainless steel substrate.

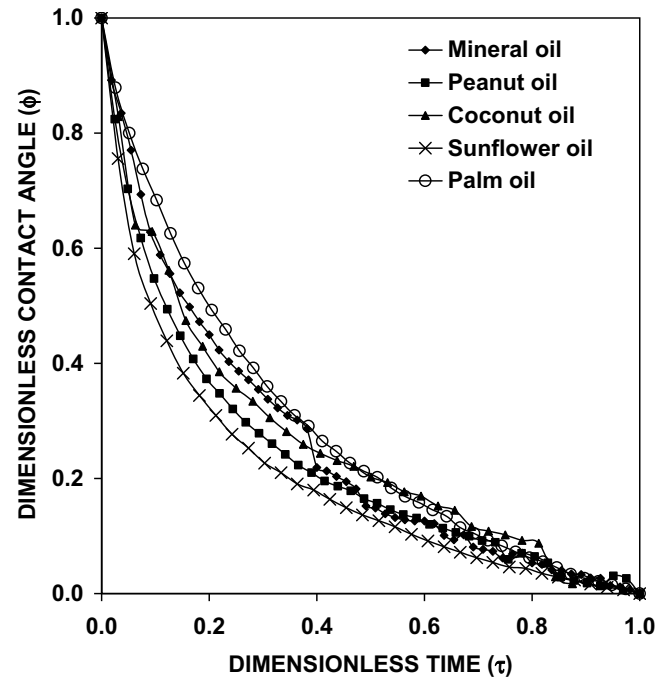


Fig. 3. ϕ versus τ plots for the spreading behaviour of oils on a smooth substrate.

other hand, as the roughness of the surface increased, the exponent shifted towards higher value. Coconut and sunflower oils, having comparable viscosities ($\mu_{\text{coconut}} = 0.0364 \text{ Pa s}$; $\mu_{\text{sunflower}} = 0.0307 \text{ Pa s}$), showed almost similar values of k (11 each for spreading on a $0.50 \mu\text{m}$ rough surface; 9 each for spreading on a $1.00 \mu\text{m}$ rough surface). The exponent ‘ n ’ was found to be sensitive to the material surface texture alone whereas the constant k is affected by both the surface texture and properties of the spreading liquid.

It is not possible to compare the spreading behaviour of various oils on surfaces with varying roughness in terms of relaxation of absolute contact angle because the rate and extent of wetting are significantly different not only for different liquids but also for different roughnesses. Hence, two dimensionless parameters ϕ and τ are used to represent contact angle and time variables respectively in order to arrive at a meaningful comparison. These two parameters are defined as follows:

$$\text{Dimensionless contact angle, } \phi = (\theta - \theta_r) / (\theta_i - \theta_r), \quad (4)$$

where θ_i is the initial contact angle, θ is the dynamic or time dependent contact angle and θ_r is the reference contact angle (the value of θ beyond which $d\theta/dt$ is $\leq 0.01^\circ/\text{ms}$)

$$\text{Dimensionless time, } \tau = (t/t_r), \quad (5)$$

where t_r is the time taken for the drop to evolve from θ_i to θ_r on a substrate and t is the transient time.

Fig. 3 shows the spreading behaviour of various oils on a smooth ($R_a = 0.25 \mu\text{m}$) stainless substrate in terms of dimensionless parameters ϕ and τ . A better understanding of relaxation behaviour of oils with time is given by noting the time taken for each of the oil for a known contact angle relaxation. Fig. 4 is a plot of relaxation time as a function of specified percent of contact angle relaxation for various oils. It is evident that coconut and sunflower oils showed rapid flow behaviour whereas kinetics of spreading of mineral and palm oils were slower. Peanut oil showed an intermediate spreading behaviour. All the oils have equal density values ($0.9 \pm 0.02 \text{ g/cc}$) and equal quantity of oil drops were dispensed during the experiments. Therefore, the gravity effect during spreading should be identical. However, the viscosity values for these oils are significantly different (Table 1). Coconut and sun-

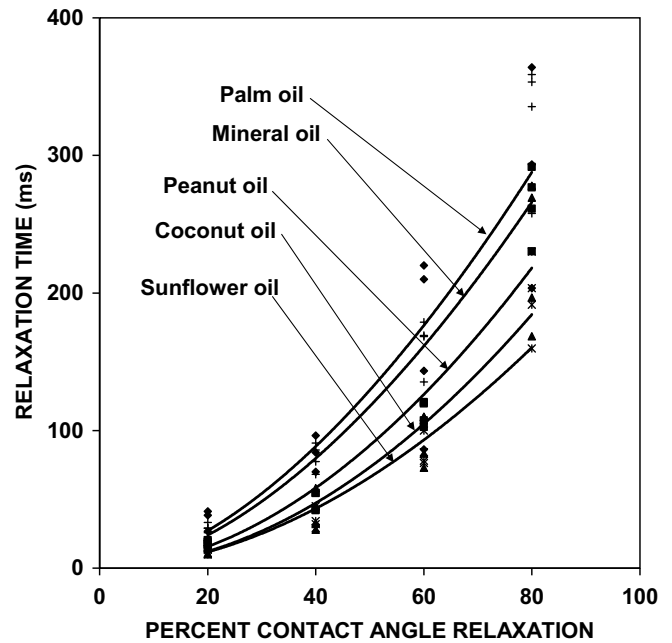


Fig. 4. Relaxation time for specified contact angle relaxations for various oils.

flower oils have low viscosities and therefore spread faster. On the other hand, viscosities of palm and mineral oils are high and comparable resulting in slow spreading.

Surface roughness had a significant effect on the kinetics of spreading. It is observed that as the roughness increases the time for relaxation also increases. Mineral oil took only 12 ms to relax 20% from a dimensionless contact angle of 1–0.8 on a smooth surface ($R_a = 0.25 \mu\text{m}$) whereas the time taken by the same oil for the same amount of relaxation on surfaces having roughness 0.50 , 1.00 and $2.00 \mu\text{m}$ were 27, 39 and 41 ms, respectively. The behaviour of

vegetable oils was also similar. Figs. 5–7 show the images captured during spreading of mineral, coconut and palm oils on stainless steel substrates of varying roughness. The dynamic contact angles at various times are indicated.

The spreading behaviour of various oils on smooth and rough surfaces consisted of different regimes. All spreadings had an initial capillary regime followed by a gravity regime. According to Cazabat et al. different regimes could be identified from the value of exponent in their behaviour $R \propto t^n$ and n had the values $1/10$ and $1/8$ in capillary and gravity regimes, respectively, in their experiments of spreading of silicone oil on hydrophilic glass substrates

[14]. However, the values of exponent obtained in the present investigation could not be used to differentiate the occurrence of capillary and gravity regimes. On the other hand, the change of slope in the $\ln(R)$ versus $\ln(t)$ plot could be conveniently used to distinguish the various regimes. Fig. 8 is a plot of logarithm of spread radius (R) versus logarithm of time (t) for mineral, coconut and palm oils during spreading on smooth ($R_a = 0.25 \mu\text{m}$) surface. A comparison of the spreading behaviour indicated significant difference between coconut and other oils. Viscous forces dominated spreading of mineral and palm oil as compared to that of coconut oil. Mineral and palm oils, having high viscosity, exhibited a well-

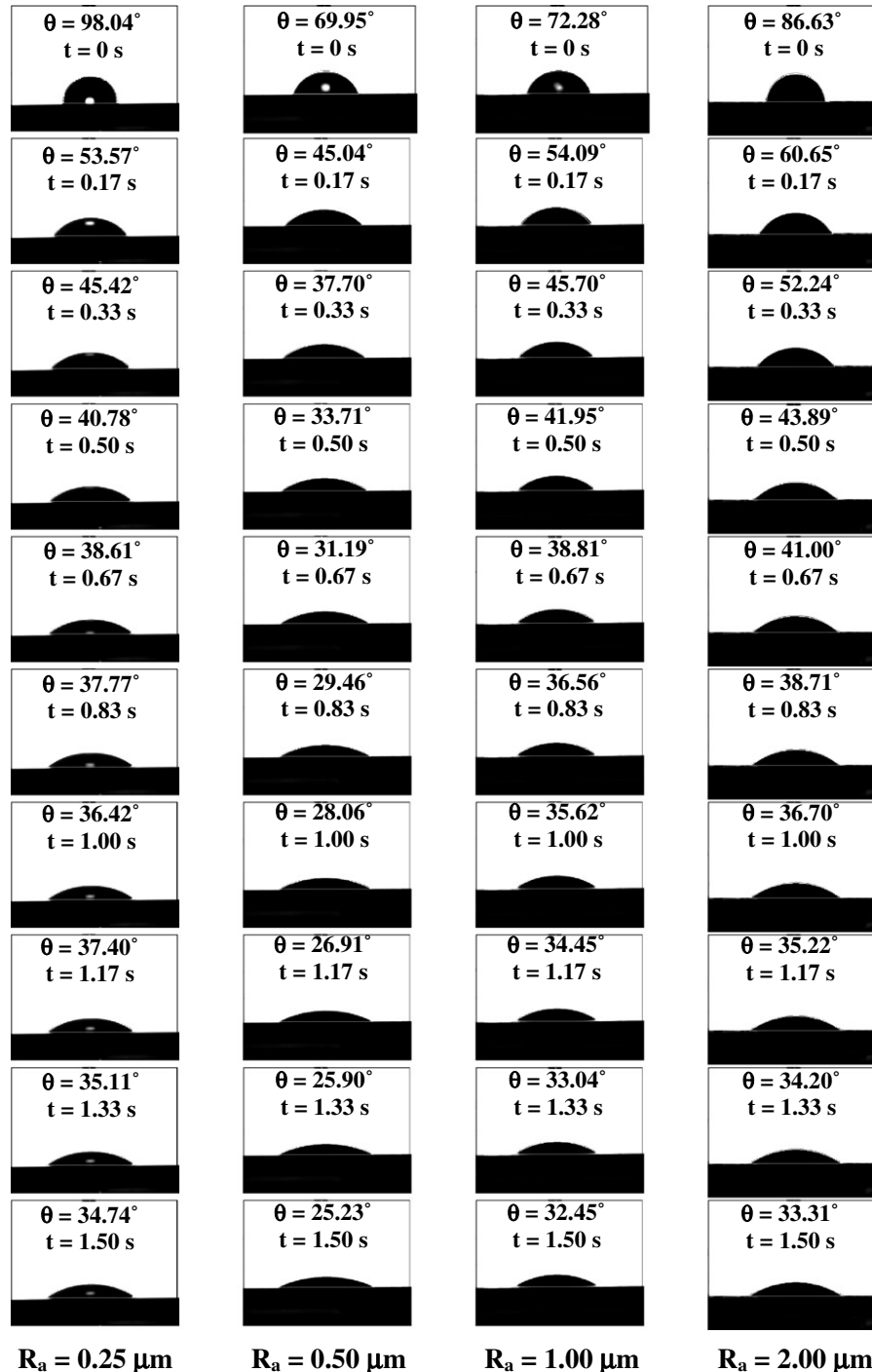


Fig. 5. Images showing the effect of substrate surface texture on spreading of mineral oil on stainless steel substrate.

defined viscous regime indicating the contact angle relaxation was almost complete. On the other hand, the low viscous coconut oil showed spreading even after 3500 ms. Hence, during spreading of low viscosity oils on smooth surfaces, longer periods of time are generally required to achieve equilibrium.

As the real surfaces are not ideally smooth, the contact angle obtained as above does not represent the intrinsic or Young's equilibrium contact angle. Fig. 9 shows the dynamic spreading of palm oil on stainless steel substrates having roughness 0.25 μm and 2.00 μm . The plot is similar in the cases of other oils also. Increasing roughness shifted the spreading curves towards slower rates of

spreading. This indicated that the rough surface texture influenced the kinetics of spreading. The spreading oil has to overcome the asperities of a rough surface which requires larger driving force. As a result rough surfaces decrease the rate of spreading and cause the spreading to extend over longer periods of time. Hence, contact angle relaxation for oils extends longer on rough surfaces before attaining equilibrium. Also, the movement of oils with higher viscosity was hindered to a greater extent compared to the oil with lower viscosity.

Fig. 10 shows the plot of $\ln(R)$ versus $\ln(t)$ for mineral, coconut and palm oils on substrates of roughness 2.00 μm . It is clearly seen

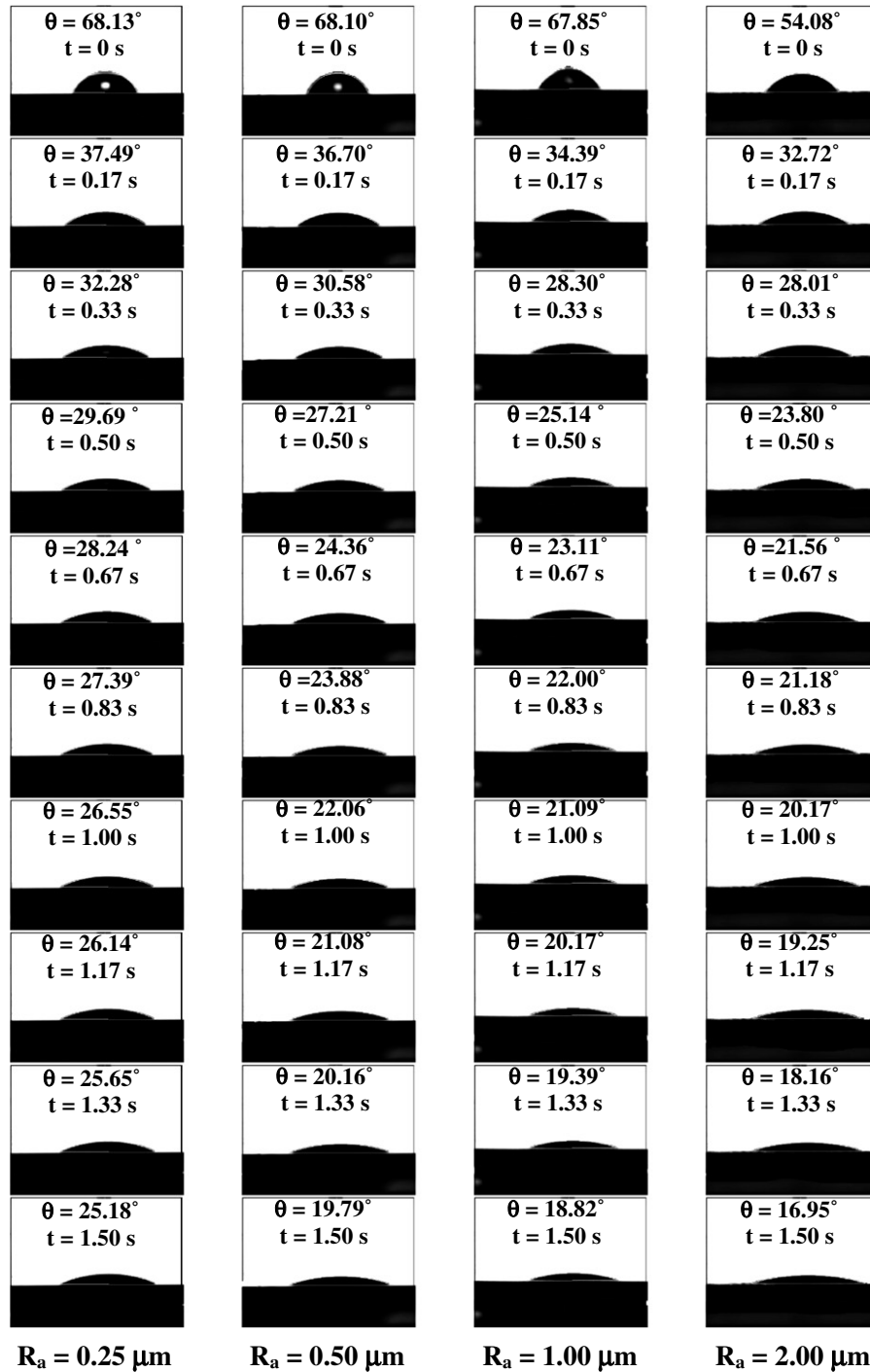


Fig. 6. Images showing the effect of substrate surface texture on spreading of coconut oil on stainless steel substrate.

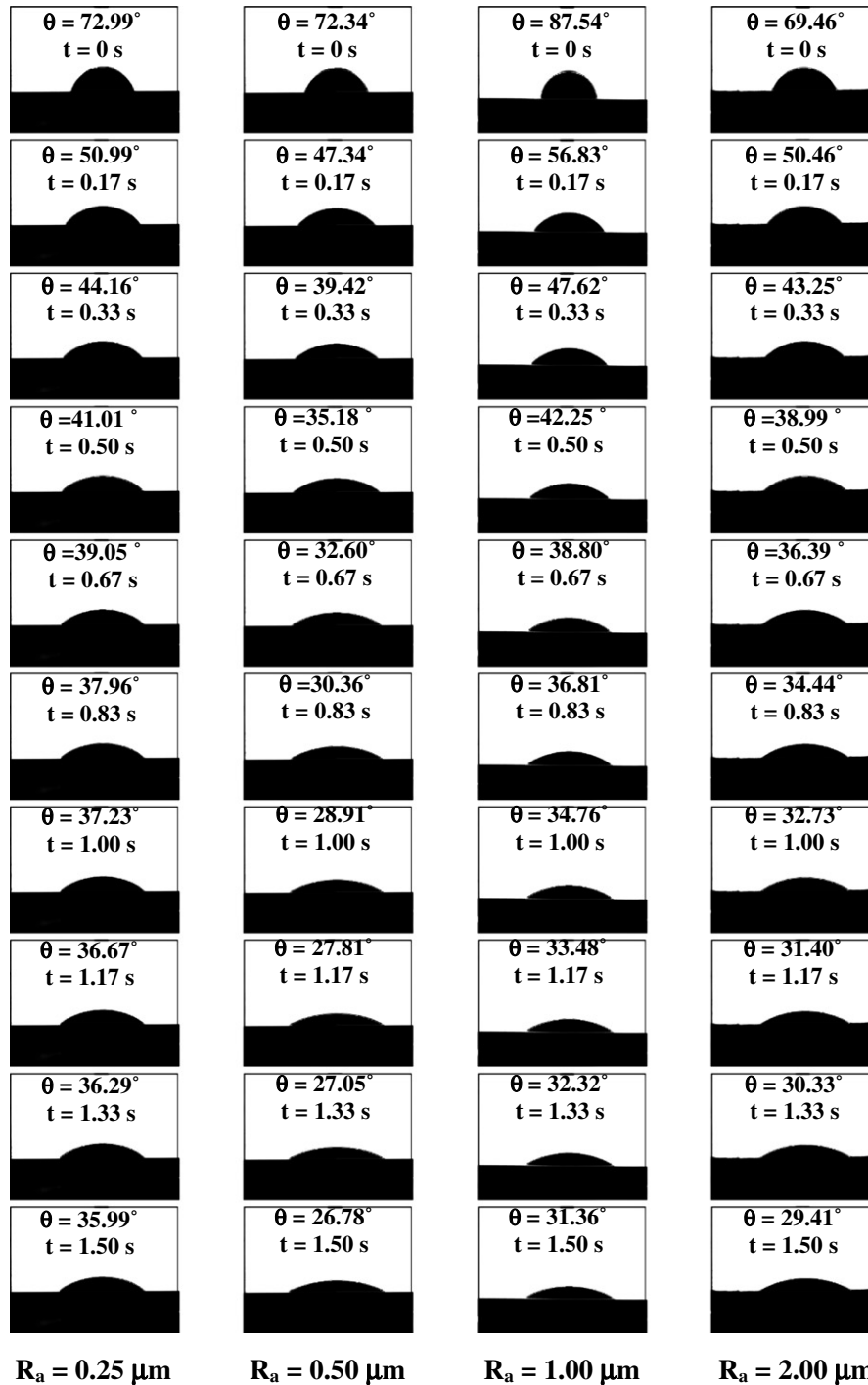


Fig. 7. Images showing the effect of substrate surface texture on spreading of palm oil on stainless steel substrate.

that at higher roughness of the substrate surface, the spreading did not terminate early on a rough substrate as compared to that on a smooth surface. For all oils, the viscous regime came into existence on a smooth surface which is indicated by decreasing slope of the curve. The slope of the curve abruptly changed as the curve became more or less horizontal while spreading on a surface with R_a value of $0.25 \mu\text{m}$. However, on rough surfaces the spreading was still in the gravity regime with nearly a constant slope.

A method is proposed here to estimate the quasi-static contact angle [18] which can be used as apparent contact angle on real surfaces for analyzing kinetics of spreading. The wetting curve of a

non-reactive liquid on an insoluble solid surface consists of two near-linear regions, viz., (i) the initial fast spreading region and (ii) region near the end of spreading period. In the case of ideal spreading, the second region is generally parallel to abscissa. On the other hand, for a real surface spreading curve is slightly inclined. Tangents are drawn to the linear portions of both the regimes so as to intersect at a point. The value of x -coordinate corresponding to this intersection point is taken as a measure of angle of contact between oil and substrate. For a smooth, ideal surface the angle corresponds to the equilibrium contact angle. Table 2 gives the contact angles determined from the spreading curve in

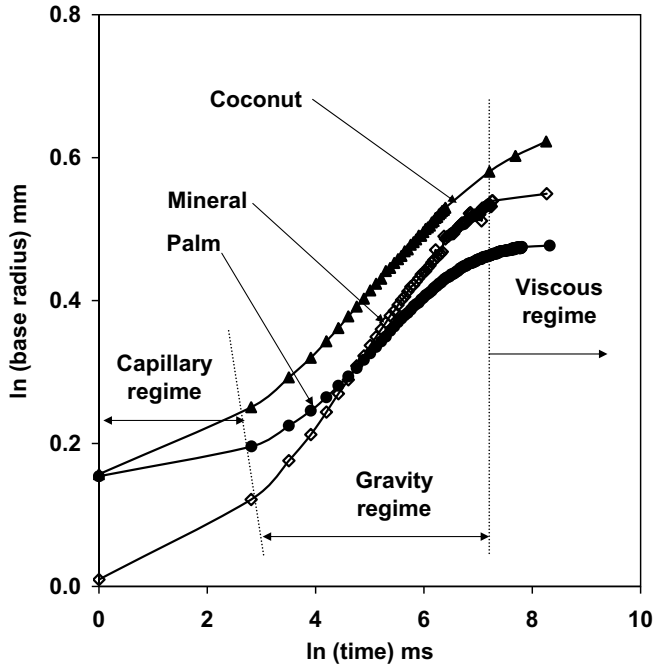


Fig. 8. Behaviour of mineral, palm and coconut oils on a smooth substrate showing different regimes.

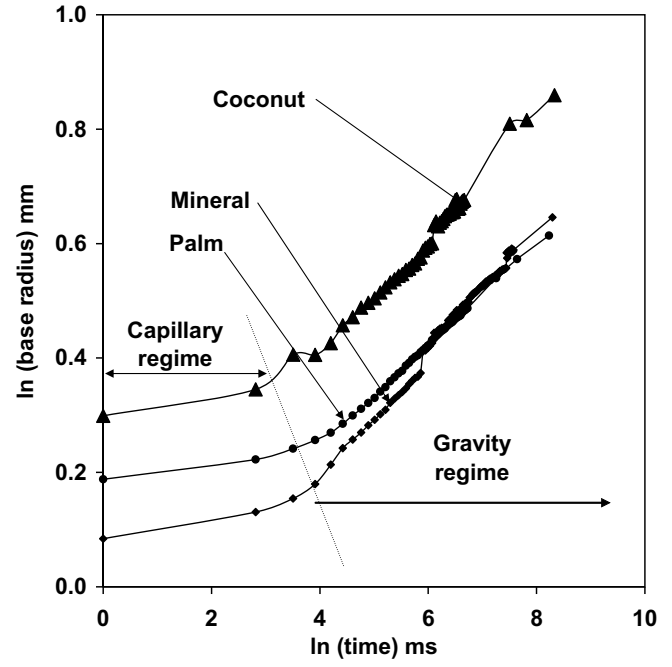


Fig. 10. Behaviour of mineral, palm and coconut oils on a rough substrate showing different regimes.

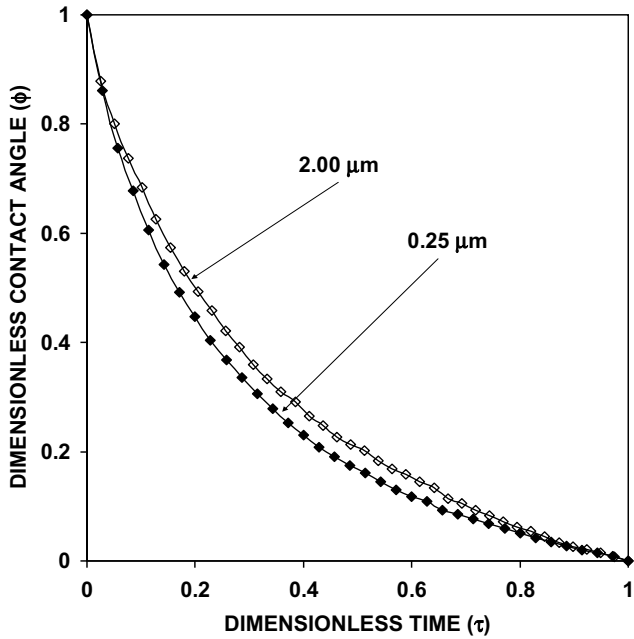


Fig. 9. Effect of surface texture on ϕ versus τ plots during spreading of palm oil.

the manner explained above under varying roughness and oil media. The data clearly shows that with increase in the roughness of the substrate surface the quasi-static contact angle decreases. For example, the quasi-static contact angle for the spreading of mineral oil on a smooth surface ($R_a = 0.25 \mu\text{m}$) was 36.5° whereas contact angles for rougher surfaces were lower: 28° , 26° and 22° on surfaces having R_a values $0.5 \mu\text{m}$, $1.00 \mu\text{m}$ and $2.00 \mu\text{m}$, respectively. A similar trend is observed during the spreading of vegetable oils as well. This is in complete agreement with Wenzel's proposition [1,4,6,8–11,14,15]. A rough surface provides an addi-

Table 2
Quasi-static contact angles under various experimental conditions

Oil medium	Roughness of the stainless steel substrate (R_a), μm			
	0.25	0.50	1.00	2.00
Mineral	36.5	28	26	22
Peanut	34	26	28	27
Coconut	26	19.5	19	16
Sunflower	33.5	27	24	18
Palm	36	28.5	25	24

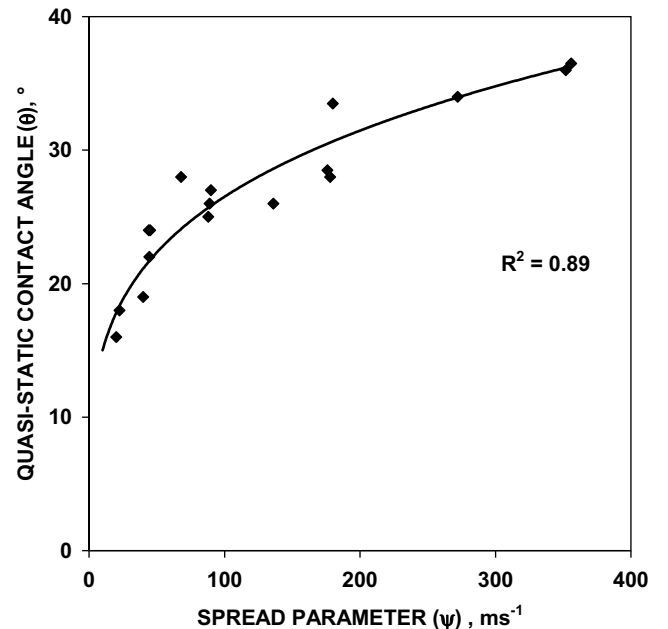


Fig. 11. Variation of quasi-static contact angle (θ) with spread parameter (ψ).

tional interfacial area for spreading liquid and thereby lowers its surface tension resulting in decrease of contact angle. It was observed that the decrease in contact angle was significant for the increase in roughness from 0.25 μm to 0.50 μm for oils. However, the effect was negligible for further increase in roughness particularly for high viscosity oils. It is likely that a critical value of roughness may exist beyond which the movement of oil is hindered owing to the physical dimensions of asperities. The increasing height/depth of the peak/valley of the substrate surface offers resistance to the spreading of the viscous liquid.

To investigate the effect of intrinsic properties of the liquid and surface texture on the evolution of contact angle, a spread parameter ψ is defined as given below:

$$\psi = \frac{\nu}{R_a}, \quad (6)$$

where ν is the kinematic viscosity of the fluid and R_a is the average value of departures of a rough surface from the mean value. The proposed parameter has the dimensions of velocity [LT^{-1}]. Fig. 11 is a plot of variation of quasi-static contact angle (θ) as a function of ' ψ '. The variation of contact angle with the spread parameter could be described by the best fit equation:

$$\theta = 8.49(\psi)^{0.25}. \quad (7)$$

A high value of roughness of the substrate coupled with low kinematic viscosity of the spreading liquid improves wetting of the substrate by the liquid. Higher roughness provides additional surface area in the form of crests and valleys for spreading whereas low viscosity facilitates efficient penetration of these asperities by the spreading liquid resulting in the evolution of lower contact angles.

The stabilized contact angle calculated by Eq. (7) was used to assess dynamic contact angles in the following manner. The wetting of a liquid on an insoluble, rigid solid is governed by power relation, $\theta = kt^n$, between the contact angle and time. Based on the results of contact angle versus time profiles in the present work, the magnitude of exponent ' n ' was modeled as a power function of surface roughness. The best fit equation is $n = -0.252(R_a)^{-0.22}$. The correlation coefficient for this equation is 0.89. The present investigation revealed that the contact angle stabilized at a time of about 3 s.

By substituting the value of ' n ', quasi-contact angle and ' $t = 3$ s in the power law equation, the magnitude of ' k ' was determined. From the knowledge of ' n ' and ' k ', the variation of contact angle with time was estimated. The empirical model was validated by experimentally measuring as well as estimating the variation of contact angle with time for spreading of castor oil on stainless steel substrates having surface roughness 1 μm . This oil was not used in developing the empirical model. Fig. 12 shows that the simulated and experimentally measured contact angles for castor oil are in good agreement. However the model is valid only for non-reactive interfaces like those used in the present work.

4. Conclusions

1. The relaxation of vegetable oils during spreading was rapid in the early stages and became gradual as equilibrium is approached. The spreading behaviour exhibited power law of the type: $A = kt^n$, where A is the drop base contact area, t is the spreading time, k is a constant and n is the exponent. The values of k and n were in the range 8–12 and 0.15–0.2, respectively, for most of the experiments carried out.
2. Low viscosity coconut and sunflower oils showed rapid spreading kinetics whereas mineral and palm oils having high viscosity exhibited slower spreading. The peanut oil showed intermediate behaviour.
3. Viscous regime for mineral and palm oils was attained earlier than that in coconut oil during spreading.
4. The time for relaxation increased with increase in roughness. Contact angle relaxation for various oils extended over longer periods before attaining equilibrium on rough surfaces.
5. Quasi-static contact angle for all the oils under investigation decreased with increasing roughness. The effect was significant for the increase in roughness from 0.25 μm to 0.50 μm for all oils.
6. A spread parameter (ψ) is proposed to account for the variation of contact angle with surface roughness of the substrate and momentum diffusivity of the spreading liquid. The quasi-static contact angle decreased with decrease in ψ according to the equation: $\theta = 8.49(\psi)^{0.25}$. An empirical model has been suggested for estimation of dynamic contact angles.

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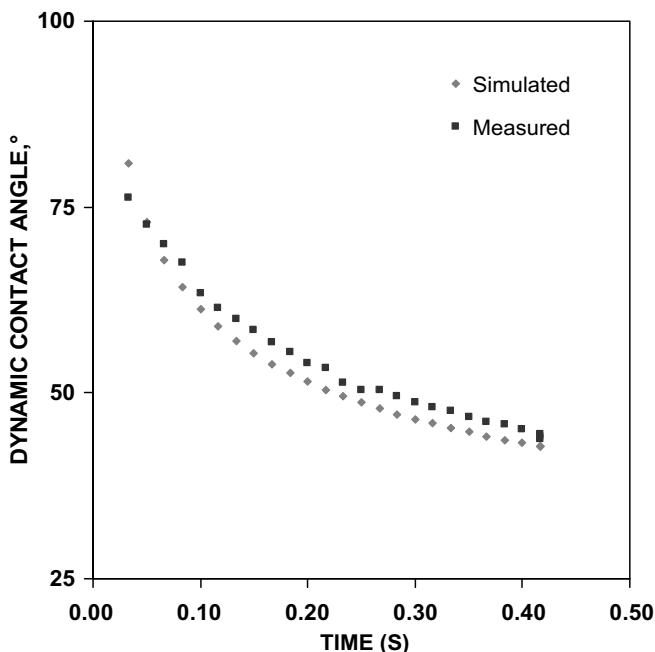


Fig. 12. Simulated and experimentally measured contact angles for castor oil.

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