Int. J. Heat Mass Transfer. Vol. 10, pp. 733–736. Pergamon Press 1967. Printed in Great Britain

THE MEASUREMENT OF CONTACT ANGLES UNDER EQUILIBRIUM AND MASS-TRANSFER CONDITIONS

A. B. PONTER, G. A. DAVIES, W. BEATON and T. K. ROSS
Chemical Engineering Department, University of Manchester Institute of Science & Technology, Manchester

(*Received* 5 *October* 1966 *and in revised form* 24 *October* 1966)

Abstract—Methods for determining contact angles between a liquid droplet on a solid surface in the presence of vapour both in equilibrium with the liquid and under mass-transfer conditions are reported. Angle values are presented for the systems methyl alcohol–water and n-propanol–water. The results suggest that the contact angle plays a predominant role in the stability of liquid films under distillation conditions.

THE IMPORTANCE of contact angle in understanding the phenomenon of liquid film breakdown under heat- or mass-transfer conditions has recently been described [1, 2]. Methods for determining contact angles under equilibrium conditions and under mass-transfer conditions at total reflux are described here. Results for the systems methanol–water and n-propanol–water on a graphite surface are presented.

1. CONTACT ANGLE OF A LIQUID IN EQUILIBRIUM WITH THE SURROUNDING VAPOUR

The apparatus illustrated in Fig. 1 consists of a 1-l. still, a cell mounted vertically above the still in which the graphite specimen is mounted, and a condenser. The still is fitted with a thermometer pocket and a condensate return line. The cylindrical cell (15 cm × 10 cm O.D.) is split into two sections, the bottom section being fitted with three optical flats. The graphite specimen is supported on a horizontal glass plate above the vapour line from the still. The still and the cell are heated electrically, the power input being controlled by two Variacs. The whole apparatus is lagged with asbestos rope to minimise heat losses. The graphite surface was carefully prepared by polishing (to a surface roughness measured by a Talysurf meter of 1–2 × 10^{-6} in), degreasing and finally washing in boiling distilled water.

A period of 2–3 h was allowed for the still to reach equilibrium. A liquid sample was then removed from the still, via the thermometer pocket, with a 2 ml syringe and re-introduced into the cell to form a sessile drop on the specimen surface. Sufficient liquid was added to the drop to establish an equilibrium drop height [3]. The drop was then allowed to come to equilibrium with the surrounding vapour in the cell. The contact angle θ was then measured by observation through a travelling microscope using a goniometer eyepiece. This procedure was repeated several times for the same liquid composition and an average value for θ taken. These values were reproducible to $\pm 1°$. Graphs of contact angle vs. liquid composition for the systems methanol–water, and n-propanol–water are shown in Figs. 3 and 4.

2. CONTACT ANGLE OF A LIQUID UNDER CONDITIONS SIMULATING TOTAL REFLUX

The apparatus for this is shown in Fig. 2. To simulate total reflux conditions a glass wetted wall column 20 cm × 6 cm I.D. was mounted above a 1-l. still. The vapour was condensed in a water cooled glass condenser and refluxed back to the column as a film. The graphite specimen was mounted horizontally in the vapour space immediately opposite a liquid sample line positioned in the wall of the column. A capillary tube was sealed into the specimen

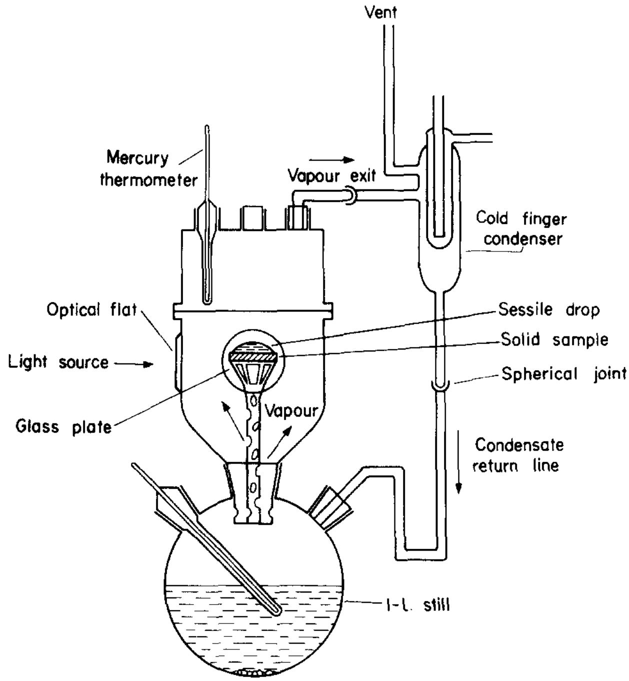

FIG. 1. Contact angle measurement under equilibrium conditions.

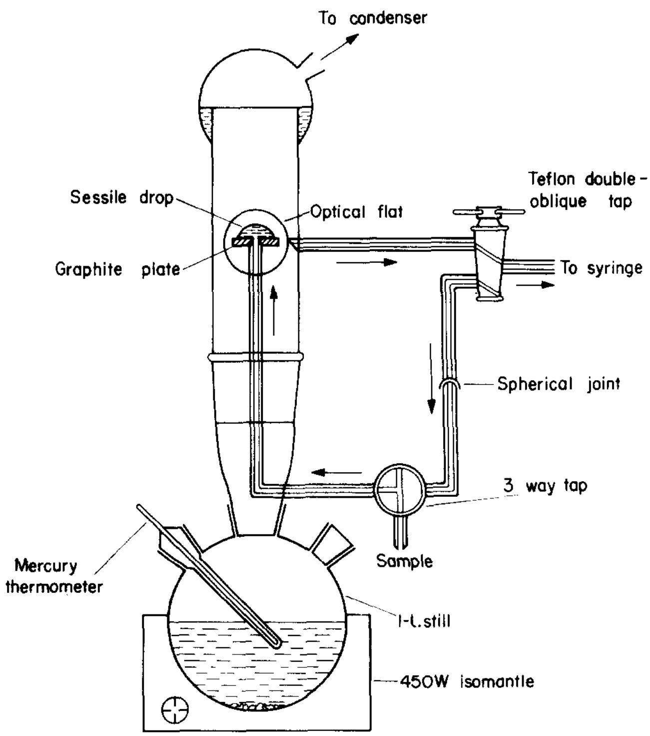

FIG. 2. Contact angle measurement under total reflux condition.

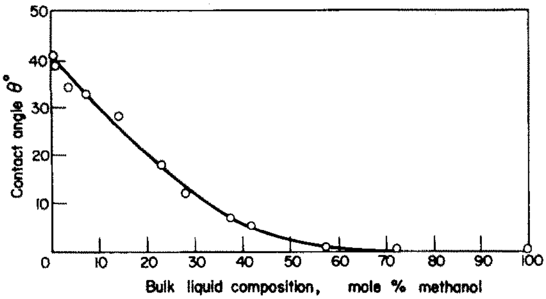

FIG. 3. Equilibrium contact angles for methanol–water–graphite system.

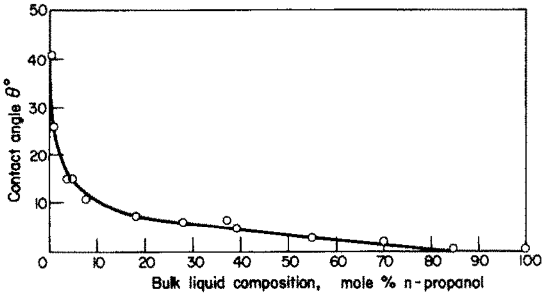

FIG. 4. Equilibrium contact angles for n-propanol–water–graphite system.

surface. The sample liquid was introduced onto the specimen through this tube. Optical flats were fitted into the column either side of the specimen. The column was fitted with an "isotape" and the whole apparatus lagged with asbestos rope. The graphite surface was carefully prepared as described previously.

The boil up rate of the still was adjusted and

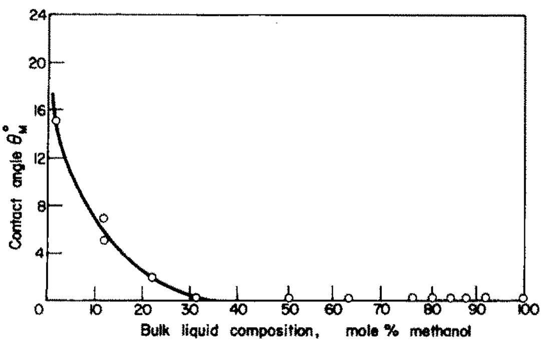

FIG. 5. Contact angles measured under total reflux conditions for methanol–water–graphite system.

the apparatus allowed to reach equilibrium. A liquid sample was then removed from the liquid film and introduced onto the specimen surface. An equilibrium drop height was again attained before the contact angle was measured. This was repeated several times for each liquid concentration as before. These values were again reproducible to $\pm 1°$. The contact angles for a range of liquid compositions for methanol–water and n-propanol–water are shown in Figs. 5 and 6.

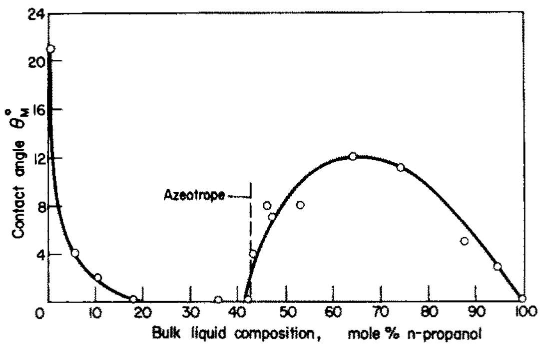

FIG. 6. Contact angles measured under total reflux conditions for n-propanol–water–graphite system.

3. DISCUSSION

The results show that the contact angle is altered when mass transfer takes place between vapour and liquid (cf. Figs. 3–5, 4–6). The results may be used to interpret the behaviour of minimum wetting rate in the system n-propanol–water.

Norman [4] has shown, when studying the minimum wetting rate of n-propanol–water mixtures in an annular wetted wall column under mass-transfer conditions, that for pure water in equilibrium with its vapour the minimum wetting rate was 12 lb/ft h. For 5% n-propanol the minimum wetting rate was reduced to about 1 lb/ft h but as the concentration of n-propanol approached the azeotropic composition (43% n-propanol) the minimum wetting rate rose sharply. It continued to rise above the azeotropic composition and at compositions between 70% and 90% n-propanol the minimum wetting rate was so high that it was impossible to maintain a continuous liquid film. Above 90% n-propanol the minimum wetting rate decreased again to a final value of 12 lb/ft h for pure n-propanol. This behaviour can be interpreted from the dependency of contact angle with concentration under mass-transfer conditions presented in Fig. 6 where after an initial reduction in contact angle θ a rapid increase in θ was observed around the azeotropic composition. At concentrations of n-propanol $>67\%$ the contact angle decreased being zero for pure n-propanol.

These results again demonstrate the importance of contact angle in assessing underwetting in transfer equipment.

REFERENCES

1. W. Murgatroyd, The role of shear and form forces in the stability of a dry patch in two-phase film flow, *Int. J. Heat Mass Transfer* **8**, 297 (1965).
2. A. B. Ponter, G. A. Davies, P. G. Thornley and T. K. Ross, The influence of mass transfer on liquid film breakdown, *Int. J. Heat Mass Transfer* **10**, 349 (1967).
3. J. F. Padday, A new method for measuring the spreading coefficient of a liquid on a solid surface, in *Second International Congress of Surface Activity*, Vol. 3, p. 136. Butterworths, London (1957).
4. W. S. Norman and D. T. Binns, The effect of surface tension changes on the minimum wetting rates in a wetted-rod distillation column, *Trans. Instn Chem. Engrs* **38**, 294 (1960).

Résumé—On décrit des méthodes de détermination d'angles de contact entre une gouttelette liquide et une surface solide en présence de vapeur en équilibre avec le liquide ou avec un transport de masse. Les valeurs des angles sont présentées pour les systèmes, alcool méthylique–eau et n-propanol–eau. Les résultats suggèrent que l'angle de contact joue un rôle prédominant dans la stabilité des films liquides pendant la distillation.

Zusammenfassung—Es wird über Methoden berichtet, den Randwinkel eines Tropfens auf fester Unterlage bei Anwesenheit eines Dampfes zu bestimmen, sowohl im Gleichgewicht als auch bei gleichzeitigem Stofftransport. Messwerte der Randwinkel werden wiedergegeben für die Mischungen Methylalkohol/Wasser und n-Propanol/Wasser in verschiedenen Konzentrationen. Aus den Ergebnissen lässt sich schliessen, dass der Randwinkel eine entscheidende Rolle bei der Stabilität eines verdampfenden flüssigen Filmes spielt.

Аннотация—Описывается методика определения контактных углов капель жидкости на твердой поверхности в процессе тепло-и массообмена, когда пар находится в состоянии равновесия со своей жидкостью. Величины углов приводятся для систем метиловый спирт–вода и *n*-пропанол–вода. Результаты свидетельствуют о том, что контактный угол играет главную роль в устойчивости жидкой пленки при дистилляции.