gators (1, 6). This is believed explainable largely by the higher rate of agitation used in this investigation.

Effect of higher moisture content in the single test made on .015-in. raw flakes in 0% miscella was to slow down the extraction rate and extraction efficiency and to raise the residual lipid level to which extraction proceeded, as would be expected. (See the first two columns under 0.015-in. raw flakes in Figure 6.)

Chemical analysis of the "total extractables" (Table V) shows that hexane extracted considerably more non-oil lipid material (phosphatides, gossypol, etc.) from the raw than from the cooked flakes, which doubtless accounts for the higher percentage of total extractables content in the raw flakes. Since these materials are known to affect adversely the oil loss and the color upon alkali refining, the total extractables or crude oil from the raw flakes, although in higher yield, would be considered of lower quality than that from the cooked flakes.

In speculating as to effect of miscella concentration on the degree of extractability, while the scope of this work is not sufficiently extensive to warrant any hard and fast conclusions, the fact that with the cooked flakes lower levels of residual lipids with increasing miscella concentration are consistently reached indicates that in addition to the solubility and diffusional factors, probable wetting ability, and the penetrating power of the extracting miscella to establish an equal concentration of extractables within the solids matrix must figure in any theoretical consideration.

The 60-min. co-current method carried out according to the procedure described above is suggested as a control laboratory method in filtration-extraction practice readily to test the comparative intrinsic extractabilities of two materials. The batch co-current method, aside from its specific applicability for determining the intrinsic extractability of variously prepared oil-bearing materials, is a valuable adjunct to the bench-scale method and apparatus (4) presently used as an evaluation unit in connection with the filtration-extraction process. The bench-scale method integrates the percentage of residual lipids (after counter current washing) with factors such as contact time, temperature, solvent ratio, cake thickness, mass velocity, solvent hold-up of cake, filter media, and quality of oil and meal products.

Summary

A batch co-current laboratory method for measuring comparative extraction rates and extraction efficiencies of oleaginous materials in solvent is described. The method, a modification of that by Winward and Shand, was carefully tested with raw and cooked cottonseed flakes of various thicknesses and in various hexane miscella concentrations. It enables measurement of intrinsic extraction rates and extractabilities of materials, unaffected by diffusional effects in the liquid medium, and yields accurate and concordant results even with extracting miscellas of considerably high concentration. It is equally applicable for evaluating and predicting the effect upon extractability of different material preparation operations, particle sizes, moisture contents, temperature, solvents, etc.

The method was used in this investigation to compare the rate and degree of extraction under the specified testing conditions of raw and cooked cottonseed flakes of .005-in., .015-in., and .025-in. thicknesses in miscella concentration of 0%, 25%, and 50% oil. The results may be summed up as follows:

a) the extractability of both raw and cooked flakes in each of the miscella concentrations decreases as the flake thickness increases.

b) the cooked material prepared from the medium and thick flakes extracted at a more rapid rate and to a greater degree in all miscella concentrations than the raw flakes of comparative thicknesses, but the rate and degree of extraction were about equal for the very thin flakes.

c) the effect of increasing miscella concentration for both the raw and the cooked flakes of medium and thick sizes was to slow down the initial extraction rate; but for the very thin flakes the effect was negligible.

d) the effect of increasing miscella concentration in extracting the cooked material, regardless of flake thickness, was to increase the degree of extraction. For the raw flakes the effect was to increase the degree of extraction only of the very thin flakes.

Acknowledgment

The authors are indebted to the Planters' Oil Mill, Greenwood, Miss., for furnishing the cottonseed used in this investigation; to Nestor B. Knoepfler for preparation of the cooked flakes; and to George I. Pittman for preparation of the drawings. The authors are also grateful to Evald L. Skau for his helpful suggestions and advice.

REFERENCES

1. Coats, H. B., and Karnofsky, G., J. Am. Oil Chemists' Soc., *27*, 51–3 (1950).
2. D'Aquin, E. L., Vix, H. L. E., Spadaro, J. J., Graci, A. V. Jr., Eaves, P. H., Reuther, C. G., Molaison, L. J., McCourtney, E. J., Crovetto, A. J., Gastrock, E. A., and Knoepfler, N. B., Ind. Eng. Chem., *45*, 247–54 (1953).
3. Fan, H. P., Morris, J. C., and Wakeham, H., Ind. Eng. Chem., *40*, 194–9 (1948).
4. Graci, A. V. Jr., Spadaro, J. J., Paredes, M. L., D'Aquin, E. L., and Vix, H. L. E., J. Am Oil Chemists' Soc, *32*, 129–131 (1955).
5. Karnofsky, G., J. Am. Oil Chemists' Soc., *26*, 564–9 (1949).
6. King, C. O., Katz, D. L., and Brier, J. C., Trans. Am. Inst. Chem. Engrs., *40*, 533–56 (1944).
7. Smith, A. S., J. Am. Oil Chemists' Soc., *29*, 421–5 (1952).
8. Wingard, M. R., and Shand, W. C., J. Am. Oil Chemists' Soc, *26*, 422–6 (1949).

[Received May 13, 1955]

CORRECTION

L. H. Dunlap, Armstrong Cork Company, Lancaster, Pa., reports that an error was made in the paper entitled "Urea Adducts of Mono- and Diesters of Fatty Acids," published in the April 1955 issue of the Journal, page 227. In paragraph two of the section on Urea Extraction of Technical Glycerol Monolaurate the statement should be that "glycerol monolaurate has a saponification number of 205" (not 246).