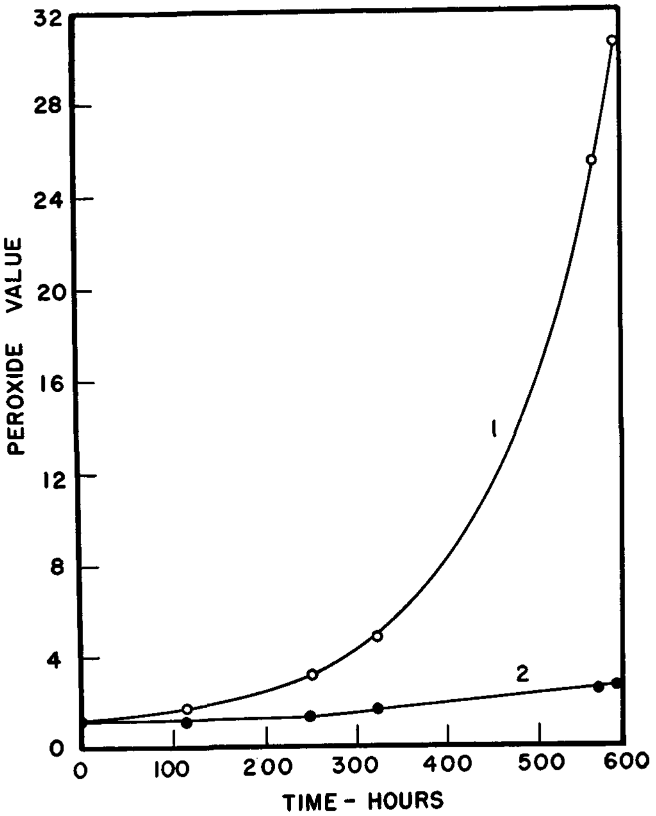

FIG. 6. Effect of dihydroquercetin and citric acid on the stability of butter oil at 60°C.
1. 0.0% antioxidant as control.
2. 0.015% dihydroquercetin and 0.015% citric acid.

these derivatives had any antioxidant activity. This confirms previous findings described in the literature that the antioxidant activity is associated with hydroxyl groups (8).

Discussion

The results of the tests on the antioxidant activity of dihydroquercetin to fats indicates that this compound is effective in the prevention of rancidity. Adequate stability of fats is accomplished with 0.03% dihydroquercetin. When used in conjunction with citric acid, an effective stability is achieved with 0.015% dihydroquercetin. Some of the findings of this laboratory were confirmed by K. F. Mattil, Research Laboratories, Swift and Company, Chicago, Illinois. He found by the active oxygen stability method that the presence of 0.05% dihydroquercetin increased the stability of lard from 5 hours to 69 hours, and he commented that this compound appears to be similar to propyl gallate in its antioxidant activity.

Dihydroquercetin is a white, crystalline, tasteless compound, which imparts no taste or color to lard and colorless oils. It has a high melting point, 252 to 253°C., and is stable at relatively high temperatures. It is easily converted into quercetin in the laboratory. A comparison of quercetin and dihydroquercetin in antioxidant activity indicated that when equal amounts (0.03%) of these substances were added to lard, dihydroquercetin was slightly less effective than quercetin. However the latter imparts a yellow color to lard.

Summary

Dihydroquercetin, a white crystalline pentahydroxyflavanone, occurring in large quantities in Douglas fir and Jeffrey pine barks, was found to be an effective antioxidant for lard, cottonseed oil, and butter oil. This compound imparts no taste and color to fats and oils and, like quercetin, appears to be nontoxic. It was found that 0.03% quercetin was slightly more effective than 0.03% dihydroquercetin as an inhibitor of rancidity in lard. The presence of a small amount of citric acid increased markedly the antioxidant effect of dihydroquercetin on lard. The pentaacetate, pentapropionate, and the pentabenzoate esters of dihydroquercetin showed no antioxidant activity.

REFERENCES

1. Bates, R. W., and Ast, H. J., J. Am. Oil Chem. Soc., *25*, 42-44 (1948).
2. Ewbank, F. C., and Gould, I. A., Oil and Soap, *19*, 205-209 (1942).
3. Fore, Sara P., Moore, R. N., and Bickford, W. G., J. Am. Oil Chem. Soc., *28*, 73-74 (1951).
4. Graham, H. M., and Kurth, E. F., Ind. and Eng. Chem., *41*, 409-414 (1949).
5. Henderson, J. L., and Young, H. A., J. Phys. Chem., *46*, 670-684 (1942).
6. Higgins, J. W., and Black, H. C., Oil and Soap, *21*, 277-279 (1944).
7. Kurth, E. F., and Kiefer, H. J., TAPPI, *33*, 183-186 (1950).
8. Mattill, H. A., Oil and Soap *22*, 1-3 (1945).
9. "Methods of Analysis of the Assoc. of Official Agr. Chemists," 6th Edition, page 501, Washington, D. C., published by A.O.A.C., 1945.
10. Report of the Committee on Analysis of Commercial Fats and Oils, October 1944, Oil and Soap, *22*, 101-107 (1945).
11. Richardson, G. A., El-Rafey, M. S., and Long, M. L., Journal of Dairy Science, *30*, 397-413 (1947).
12. Riemenschneider, R. W., and Speck, R. M., Oil and Soap, *22*, 23-25 (1945).
13. Riemenschneider, R. W., Turer, J., and Speck, R. M., Oil and Soap, *20*, 169-171 (1943).
14. Wheeler, D. H., Oil and Soap, *9*, 89-97 (1932).

[Received April 24, 1951]

Viscosity of Cottonseed Meal Dispersions

JOSEPH T. HOGAN and JETT C. ARTHUR JR., Southern Regional Research Laboratory,[1]
New Orleans 19, Louisiana

THE apparent viscosities of cottonseed meal dispersions, i.e., properties of the dispersions which offer definite resistance to change in form or flow, are important in adapting cottonseed meal for utilization as plywood glues or special sizes. One application of these data is in developing a cottonseed meal glue with a long "working life," i.e., a glue having a low viscosity for several hours, to facilitate the application of the glue to the laminate. In previous publications these properties have been reported for cottonseed meal glues and proteins (1-3, 5). The purpose of the present report is to present data on the viscosity properties of usable cottonseed meal dispersions *per se* and to compare these properties with those of cottonseed protein dispersions and cottonseed meal glues.

---

[1] One of the laboratories of the Bureau of Agricultural and Industrial Chemistry, Agricultural Research Administration, U. S. Department of Agriculture.

Experimental

Cottonseed Meal. Oil-free meal was prepared by removing the oil from flaked cottonseed meats by means of commercial hexane as described in a previous publication (4). The meal contained (on a moisture free basis) 8.98% nitrogen and 1.53% lipids. About 80% of its total nitrogen content was soluble in 0.5 M sodium chloride, as determined by suspending 2.5 grams of meal in 100 ml. of 0.5 M sodium chloride for 3 hours at 25°C. The dried meal was ground to pass a 200-mesh screen. This degree of fineness is required in the preparation of a meal glue having satisfactory spreading properties in plywood gluing operations. Since the meal which passed 200-mesh is of primary interest in making glue dispersions, the apparent viscosity of these meal dispersions was determined.

Apparatus. The apparent viscosities of the cottonseed meal dispersions were measured with a torsional wire viscosimeter (6) at a temperature of 25°C. The viscosimeter was calibrated by means of standard viscosity oils provided by the National Bureau of Standards. (The dimensions of the viscosimeter were diameter of exterior cup 3.02 cm., diameter of interior cylindrical rotor 1.26 cm., and depth of immersion of rotor in solution 4.92 cm. The exterior cup was rotated at a speed of 20 revolutions per minute.)

Results and Discussion

It has been previously shown that factors which affect the viscosity properties of cottonseed protein dispersions are the concentration of the protein, the amount of sodium hydroxide used to disperse the protein, the age of the dispersion, and the presence of specific anions (1, 2). These factors have been investigated for cottonseed meal dispersions with results as reported here.

Effect of Meal Concentration. The effect of varying the meal concentration on the apparent viscosity of dispersions containing 4.4 grams of sodium hydroxide and 1.25 grams of trichloroacetic acid per 100 grams of meal is shown in Table I. The viscosities of the meal dispersions were determined after 1, 2, and 3 hours of aging the dispersions.

TABLE I

Effect of Meal Concentration on the Apparent Viscosity of Cottonseed Meal Dispersions [a]

Cottonseed meal, g./100 g. water	pH [b]	Apparent viscosity, poises		
		1 hour	2 hours	3 hours
13	12.9	121	103	92
17	12.7	187	152	118
22	12.7	286	227	215
28	12.4	309	275	252
34	12.3	335	321	287
40	12.0	361	314	302

[a] Cottonseed meal dispersions also contain 4.4% sodium hydroxide and 1.25% trichloroacetic acid based on the meal weight.
[b] pH values were determined after aging the dispersions for 1 hour.

It is seen that the viscosities of the dispersions increase rapidly with an increase in meal concentrations below 25 grams of meal per 100 grams of water and that in meal concentrations greater than this value the viscosities increase almost linearly and less rapidly with an increase in meal concentration.

On comparing these data with similar data previously reported for isolated protein (2, 3), it is clear that the viscosities of protein dispersions are much more sensitive to changes in protein concentration than are the viscosities of meal dispersions sensitive to changes in meal concentration. These observations are true when the two types of dispersions are compared on either a solids content or a protein content basis. As the concentrations of protein and meal decrease, the viscosities of the meal dispersions are greater than the viscosities of the protein dispersions; however as the concentrations of protein and meal increase, the viscosities of the protein dispersions are greater than the viscosities of the meal dispersions. The effect of the suspended cellulosic components of the meal and the interaction of the protein contained in the meal with the ash and carbohydrate components probably account for these observed relationships.

Effect of Sodium Hydroxide Concentration. The effect of sodium hydroxide concentration on the viscosity of dispersions prepared with hexane-extracted meal was determined and is shown in Table II. An increase in sodium hydroxide concentration from 4.4 grams per 100 grams of meal to 6.6 grams per 100 grams of meal resulted in a decrease in the viscosity of the dispersions at all meal concentrations; however the change in viscosity was less at the higher meal concentrations.

TABLE II

Effect of Sodium Hydroxide Concentration on the Apparent Viscosity of Cottonseed Meal Dispersions [a]

Cottonseed meal, g./100 g. water	NaOH Conc. (g./100 g. meal)					
	4.4		5.5		6.6	
	Viscosity and pH at two hours					
	pH [b]	Poises	pH [b]	Poises	pH [b]	Poises
13	12.9	103	13.1	91	13.2	75
17	12.7	152	12.9	147	12.9	127
22	12.7	227	12.8	212	12.8	186
28	12.4	275	12.6	231	12.7	202
34	12.3	321	12.8	278	12.5	248
40	12.0	314	12.3	324	12.4	301

[a] Cottonseed meal dispersions also contain 1.25 g. trichloroacetic acid per 100 g. meal.
[b] pH values were determined after aging the dispersions for 1 hour.

On comparing these data with those for isolated protein (2, 3) it is observed that the viscosity of the protein dispersions are much more sensitive to changes in sodium hydroxide concentration than are the meal dispersions. This may be due to the stabilizing effect of the carbohydrate material contained in the meal, analogous to the effect of sugars on the viscosity of protein dispersions (1, 3).

Effect of Trichloroacetate Ion Concentration. The preparation of cottonseed protein dispersions by the

TABLE III

Effect of Trichloroacetic Acid on the Apparent Viscosity and Tackiness of Cottonseed Meal Dispersions [a]

Trichloroacetic acid, g./100 g. meal	pH [b]	Apparent viscosity and tackiness		
		1 hour	2 hours	3 hours
0.00	13.1	265 poises poor tack	242 poises poor tack	214 poises poor tack
0.55	12.9	271 poises poor tack	242 poises poor tack	216 poises poor tack
1.10	12.7	269 poises good tack	249 poises good tack	230 poises good tack
1.75	12.5	238 poises very good tack	215 poises very good tack	198 poises very good tack
2.20	12.1	210 poises poor tack	187 poises poor tack	163 poises poor tack

[a] Cottonseed meal dispersions also contain 100 parts of meal, 400 parts of water, and 5.5 parts of sodium hydroxide.
[b] pH values were determined after aging the dispersions for 1 hour.

TABLE IV

Effect of Method of Removal of the Oil and Solubility of the Protein on the Apparent Viscosity of Cottonseed Meal Glue Mixes

Meal	Nitrogen, %	Lipids, %	Percent of total nitrogen soluble[a]	pH of glue mix	Viscosity poises[b]
					(1 hr.)
Hydraulic-pressed	6.6	5.6	20.2	12.9	322
Hydraulic-pressed	6.7	5.6	39.0	12.9	297
Screw-pressed	6.8	3.6	40.0	13.2	311
Screw-pressed	6.9	3.6	42.7	12.8	317
Screw-pressed	6.9	4.7	50.4	12.7	295
Hexane-extracted	9.0	1.5	80.0	12.4	320

[a] Percentage of total nitrogen soluble in a 0.5 M sodium chloride, 2.5 grams of meal suspended in 100 ml. of solvent for 3 hours at 25°C.

[b] Glue mix compositions: Cottonseed meal, 100 parts; sodium hydroxide, 4 parts; sodium silicate, 15 parts; carbon disulfide-carbon tetrachloride, 3 parts; calcium hydroxide, 15 parts; trichloroacetic acid, 1.25 parts; water, 335 parts. See Hogan and Arthur, J. Am. Oil Chem. Soc., *28*, 20-23 (1951).

addition of trichloroacetate ion to prevent gel formation and to improve their tackiness was previously reported (1, 2). The data presented in Table III show that the presence of trichloroacetate ion has a similar effect on the properties of cottonseed meal dispersions. The optimum amount of ion to add is about 1.3 grams of trichloroacetic acid per 100 grams of meal; when compared on a protein basis, this amount is about equal to the optimum quantity added to protein dispersions. It is seen that if an amount in excess of this quantity is added to the meal dispersion, a decrease in its viscosity is observed.

Effect of Method of Removal of the Oil and Solubility of the Protein Contained in the Meal. The apparent viscosities of glue mixes, prepared from cottonseed meals from which the oil has been removed by different processes, are compared in Table IV. An indication of the extent of denaturation of the protein contained in the meal, which takes place when the oil is extracted, is its change in nitrogen solubility. In these selected meals their nitrogen solubilities in 0.5 M sodium chloride solution varied from 20 to 80% of the total nitrogen. It is significant that there is no substantial difference in the viscosities of the glue mixes prepared as indicated. However, as noted in a previous publication, a decrease in the nitrogen solubility of the protein contained in the meal results in a decrease in the adhesive strength of the glue.

On comparing these data with those for isolated protein, it is observed that the viscosities of the protein dispersions are also affected by the method of the removal of the oil from the meats (3).

Summary

It has been shown that the apparent viscosities of cottonseed meal dispersions depend on the concentration of meal in the dispersion, the concentration of sodium hydroxide in the dispersion, and the addition of trichloroacetate ion and that the viscosities of the glue dispersions are independent of the nitrogen solubilities of the protein contained in the meal and, for the processes evaluated, independent of the method of removal of the oil to produce the meal.

REFERENCES

1. Arthur, J. C. Jr., U. S. Patent 2,531,383, November 28, 1950.
2. Arthur, J. C. Jr., and Karon, M. L., J. Am. Oil Chem. Soc., *25*, 99-102 (1948).
3. Cheng, F. W., and Arthur, J. C. Jr., J. Am. Oil Chem. Soc., *26*, 147-50 (1949).
4. Gastrock, E. A., and D'Aquin, E. L., Oil Mill Gazetteer, *53*, 13-21 (1948).
5. Hogan, J. T., and Arthur, J. C. Jr., J. Am. Oil Chem. Soc., *28*, 20-23 (1951).
6. MacMichael, R. F., Ind. Eng. Chem., *7*, 961-3 (1915).

[Received February 28, 1951]

Better Oil From New or Old Deodorizers[1]

F. B. WHITE, Foster Wheeler Corporation, New York, N. Y.

THERE are at least seven factors influencing the product obtainable from a deodorizer. These are: condition of feed stock, condition of stripping steam, condition of equipment, materials of construction, method of heating, operating vacuum, and time of deodorization. Consideration must be given to all of these points in analyzing for imperfections in a deodorizing system. However it is not intended to discuss these factors in detail but to touch upon them lightly, with more emphasis upon several specific ideas which may prove helpful to a refinery operator or superintendent.

Condition of Feed Stock

To produce a high grade of deodorized product it is necessary properly to pre-condition the oil being fed to a deodorizing system. Refining and bleaching techniques are well known, and substantially complete elimination of soaps is a "must" for good deodorization. Most soaps are generally removed at the tail end of the refining operation, but subsequent bleaching is needed to insure a top quality final product. The bleaching operation does take out traces of soap which would otherwise be present after refining and which cannot be removed by deodorization.

Condition of Stripping Steam

It is important that adequate stripping steam be used and that it have sufficient superheat to avoid cooling the oil being deodorized. Any marked reduction in oil temperature will reduce the effectiveness of deodorization by reducing the vapor pressures of the undesirables to be removed. In the author's opinion this does not mean that steam must enter the deodorizer at precisely the same temperature as the oil, or for that matter above it, since the amount of sensible heat required to raise the temperature of dry steam to the deodorization temperature is quite small. However it does mean that the steam must be completely dry, contain sufficient superheat to insure dryness throughout the piping system leading to the deodorizers, and enter the deodorizer at a temperature reasonably close to that of the oil batch. From a practical point of view, in a batch deodorizer with a well designed sparger, the blowing steam temperature may be 30°F. below the oil temperature without noticeable effect because the heat transfer from the oil to the steam is accomplished over a wide area. Due also to the immense steam volume it is impossible for a particular portion of the oil body to become chilled. On the other hand, the presence of moisture from wet steam requires a concentrated application

---

[1] Presented at the 42nd Annual Meeting, American Oil Chemists' Society, New Orleans, La., May 1-3, 1951.