Device to Establish Drying Conditions for Protective Coatings at Elevated Temperatures[1]

A. W. SCHWAB, H. M. TEETER, and J. C. COWAN, Northern Utilization and Development Division, Agricultural Research Service, U. S. Department of Agriculture, Peoria, Illinois

IN THE EVALUATION of coatings prepared from vinyl ether formulations (2) it was necessary to determine drying times at elevated temperatures. A Sanderson drying-time meter (3, 4) is adequate for normal room temperatures, but apparently no similar device has been designed for higher temperature studies. The apparatus described is basically the same as Sanderson's but smaller and constructed in an oven where temperatures up to 260°C. can be maintained.

Apparatus

The oven is a standard, double-door, mechanical convection type with an adjustable air-flow control (Precision Scientific Company, Model 845, Type A, Cat. No. 1248). Temperature range is 35° to 260°C. and the total watts 5,300. Inside-cabinet dimensions for width, depth, and height are 37, 19, and 25 in., respectively. The floor space occupied is 49 x 27 in., and the over-all height is 64 in. Figure 1 pictures the oven with the apparatus mounted inside. An adjustable air-flow control regulates the air velocity across the working chamber from approximately 75 to 250 ft./min. All of the air in the working chamber is forced to travel through a complete cycle of about 50 air changes per minute.

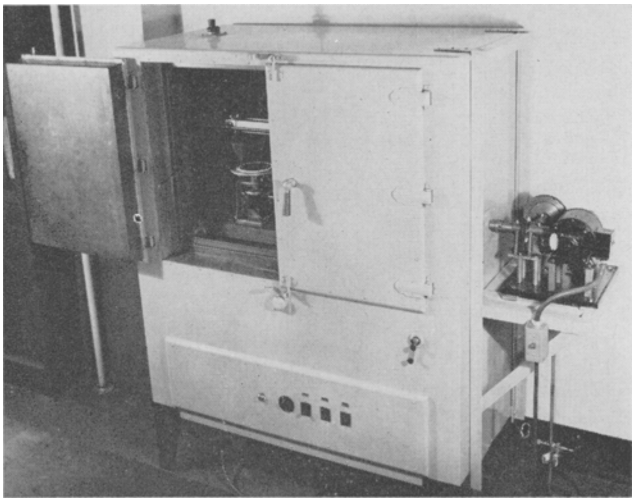

FIG. 1. Exterior view of oven showing drive mechanism on right side.

On the outside and on the right-hand side of the oven is mounted a 1½-in. angle iron frame. This frame supports a 14 x 18-in. platform, on which is located the motor, reducing gears, and drive mechanism. The motor is a ⅛-hp. Westinghouse-type FH with an r.p.m. of 1,725. This r.p.m. is reduced to approximately 0.067 (1 revolution every 15 min.) through a series of worm and bevel gears. A coupling on the inside of the oven permits removal of the inside mechanism should the occasion arise.

FIG. 2. Interior view of oven showing turntables and sanding mechanism.

The inside mechanism is also mounted on 1½-in. angle iron as shown in Figure 2. Tolerances of the order of 0.015 in. were made for gear clearances and bearing freedom to compensate for heat expansion. Alemite fittings were placed on all locations of bearing contact and are greased with a special high-temperature lubricant (Lubriplate 930 AA manufactured by the Fiske Bros. Refining Company, Newark, N. J.). The turntables were made of aluminum 7 in. in diameter. Three guideposts (approximately $^{3}/_{16}$ in. diameter) were placed in a semicircle ½ in. from the circumference of the aluminum turntable to aid in centering a 6-in. coated disc. Discs were stamped from sheet aluminum and black iron 0.023 in. thick with a 6-in. die.

Temperature checks were made throughout the oven with thermocouples, and the largest differential found in the oven proper was 2°C. Thermocouples placed under aluminum and black iron discs showed no appreciable difference in temperature that might arise from the more heat-reflective aluminum. The time required for the oven to return to temperature after opening for placement of the coated discs is approximately 5 min. at the 150°C. baking level; at 200°C. the time is 8 min. Under established procedure the oven was heated approximately 20 degrees above the

---

[1] Presented at fall meeting, American Oil Chemists' Society, Chicago, Ill., Oct. 20–22, 1958.

temperature at which the test was to be conducted. Usually by the time that the discs were mounted and the doors closed, the oven was at or near the desired temperature. The thermostat is then reset to the predetermined value.

Sixty-mesh white sand (sieved) is used in the sanding cone. This cone is 3½ in. high with a base of 3¼ in. in diameter. The opening in the cone where the sand is fed to the disc is 0.070 in. The tip of the cone is set at 0.030 in. above the coated disc and allows a sand trail approximately 0.25 in. wide. With 6-in. discs this setting permits readings up to about 2 hr. with the turntables revolving at 0.067 r.p.m. "Tack-free time" is determined by the usual method of inverting the disc, tapping sharply, and noting the point at which the sand does not adhere to the film. "Dry-to-touch-time" is indicated by the point at which the sand is still imbedded in the film after vigrous brushing with a paint brush. In interpreting the results, it should be recognized that the observed characteristics are those of the films at elevated temperatures.

Preparation of Coatings

Coatings are prepared by the spinning disc method (6). Figure 3 is a photo of the spinning apparatus. A number of factors determine the thickness of film prepared in this manner, and these have been investigated by Gardner and Sward (1). With our vinyl ether formulations most 25% solutions in toluene have a Gardner viscosity of about A-3. When a 5-g. sample of this solution is added to the center of a spinning disc at 400 r.p.m. and rotation is continued for 2 min., a film is obtained, which upon baking for 10 min. at 200°C., has a thickness of about 0.15 mil. This value is in the range at which a number of can coatings are made. The thickness is determined by cutting a measured square from the disc and weighing it. After the coating is removed by warming in ethanolamine, the disc is washed, dried, and weighed again. Assuming a density of 0.9, the thickness is calculated from the following formula: thickness (mils) = loss in wt. (g.) × 1,000/0.9 × area (cm.^{2} × 2.54.

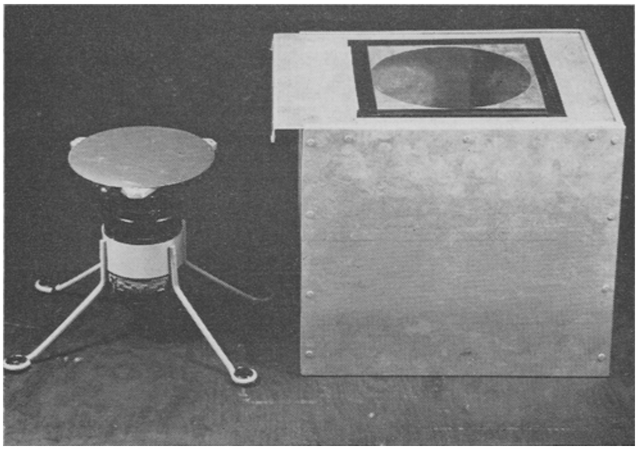

FIG. 3. Apparatus for spinning coatings.

There is no explosion hazard from the toluene present in the wet films because the total concentration of toluene in the oven is less than 0.04%, well below the lower explosive limit.

Results

Experiments conducted on discs coated with the same material and run simultaneously showed no differences in drying times. The top panel in Figure 4 illustrates the results of such an experiment. The sand trails represent the dry-to-touch time for an isobutyl-(3)-conjugated soy-(1)-vinyl ether copolymer,[2] coated on aluminum discs and baked at 150°C. without drier. A repeat run on the following day gave similar results. The bottom panel in Figure 4 shows sand trails for a third experiment of the same type except that the coating is a nonconjugated linseed vinyl ether homopolymer baked on black iron discs at 100°C. without drier. Again the dry-to-touch times are the same, and the uniformity of conditions in the oven is demonstrated.

During evaluations on black iron and aluminum it was noted that shorter drying times were encountered with black iron; this was true for both tack-free and dry-to-touch time. Table I lists the results of a study of tack-free time for an isobutyl-(3)-conjugated soy-(1)-vinyl ether copolymer on aluminum as well as black iron, and at three different baking temperatures with and without drier. In all comparable tests the black iron gave shorter tack-free times. The difference is even more pronounced in the dry-to-touch times compiled in Table II. Although the reason for faster drying on black iron is not known, it is likely that iron plays the role of an oxidation or polymerization catalyst in the drying process. To test further this hypothesis, an experiment was conducted with iron naphthenate drier (0.1% Fe based on weight of copolymer). The drier was added to the same isobutyl copolymer, and coatings were made on both aluminum and black iron. A third disc of aluminum was coated only with the isobutyl copolymer (*no* drier). The three samples were placed simultaneously on the drying-time meter at 180°C. The results of this experiment are listed in Table III and give additional evidence that iron definitely accelerates drying. Identical drying times were obtained with the coatings

TABLE I

Tack-Free Time for Films on Aluminum and Black Iron [a]

Baking temperature	Tack-free time, min.			
	Aluminum		Black iron	
	Drier [b]	No drier	Drier [b]	No drier
°C.				
150	>150	>150	120	>120
180	120	>120	50	75
210	40	40	15	15

[a] Isobutyl-(3)-conjugated soy-(1)-vinyl ether copolymer.
[b] 0.1% Cobalt as naphthenate.

TABLE II

Dry-to-Touch Time for Films on Aluminum and Black Iron [a]

Baking temperature	Dry-to-touch time, min.			
	Aluminum		Black iron	
	Drier [b]	No drier	Drier [b]	No drier
°C.				
150	25	60	16	35
180	15	25	12	15
210	7	10	1	5

[a] Isobutyl-(3)-conjugated soy-(1)-vinyl ether copolymer.
[b] 0.1% Cobalt as naphthenate.

---

[2] The (3) and (1) designate the molar amounts of respective comonomers.

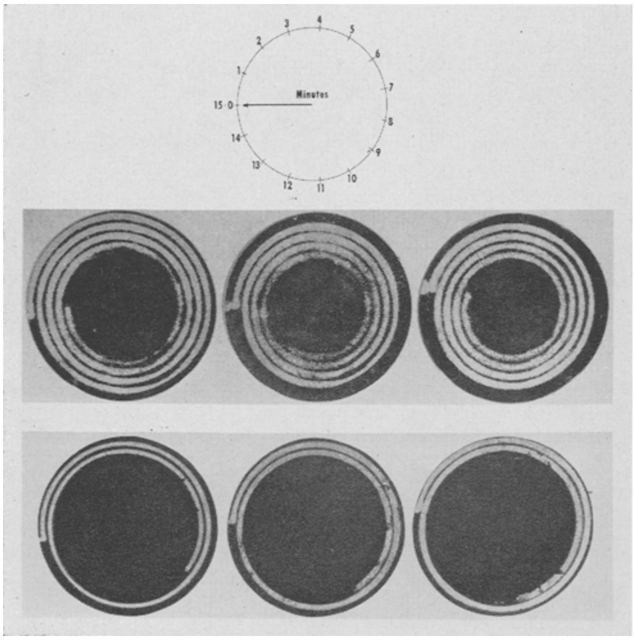

FIG. 4. Top panel—sand trails of an isobutyl-(3)-conjugated-soy-(1) copolymer baked at 150°C. without drier. Bottom panel—sand trails of a nonconjugated linseed vinyl ether homopolymer baked at 100°C. without drier.

TABLE III

Effect of Iron Naphthenate on Drying Time of Films at 180°C.[a]

	Tack-free time	Dry-to-touch time
	min.	*min.*
With drier [b] on Al	45	8
No drier on Al	>120	25
With drier [b] on Fe	45	8
No drier on Fe	75	15

[a] Isobutyl-(3)-conjugated soy-(1)-vinyl ether copolymer.
[b] 0.1% Fe as naphthenate.

containing drier; the catalytic effect of the black iron disc is masked by the presence of iron naphthenate.

A comparison of the relative effectiveness of cobalt and iron naphthenate on drying time may be made from an inspection of the data in Tables I, II, and III. The conclusion that iron naphthenate contributes to faster drying is in agreement with other observations made (5).

Summary

An apparatus has been described for determining the drying time of protective coatings at elevated temperatures, and tests have been made with vinyl ether formulations. The device is essentially a miniature Sanderson drying-time meter constructed in an oven where temperatures up to 260°C. can be maintained. Tolerances were made on the order of 0.015 in. for gear clearances and bearing expansion. A special high-temperature grease is the lubricant. Turntables, which make one revolution every 15 min., permit simultaneous evaluation of three films. Reproducible results have been obtained, and uniform conditions apparently prevail throughout the oven.

Coatings are prepared by the spinning disc method, and under controlled conditions the film thickness is approximately 0.15 mil. "Tack-free time" and "dry-to-touch time" are determined by the usual method of removing sand from the disc. Discs were stamped from aluminum as well as black iron, and differences in drying times observed. Coatings on black iron dry faster than those on aluminum, and it is presumed that iron plays the role of an oxidation or polymerization catalyst.

Acknowledgments

The authors are indebted to Alfred F. Fitton and Harold E. Ladd for the construction of the apparatus and to John A. Stolp for assistance in some of the determinations.

REFERENCES

1. Gardner, H. A., and Sward, G. G., Ind. Eng. Chem., *19*, 1972 (1927).
2. Gast, L. E., Schneider, W. J., O'Donnell, J. L., Cowan, J. C., and Teeter, H. M., J. Am. Oil Chemists' Soc., *35*, 347 (1958).
3. Sanderson, J. McE., Proc. A.S.T.M., *25*, Part II, 407 (1925).
4. *Ibid.*, *26*, Part II, 556 (1926).
5. Unpublished data.
6. Walker, P. H., and Thompson, J. G., Proc. A.S.T.M., *22*, Part II, 465 (1922).

[Received December 8, 1958]

A New Concept in Vegetable Oil Refining Automation

COLLIN M. DOYLE, HERBERT R. KAISER, and ARNOLD M. GAVIN,[1] Podbielniak Inc., Chicago, Illinois; and HARRY SIEG, Fischer and Porter Company, Hatboro, Pennsylvania

THE REFINING of vegetable oils began as an art with the kettle or batch process. Continuous refining has been made possible by the use of proportioning control for addition of reagent to crude oil, temperature control, and centrifuges for more rapid separation of oil from gum or soap stock. Plants of large refining capacity which use multiple centrifuge installations do not lend themselves however to practical automation. The lack of automation requires the attention of a skilled operator.

This paper presents our approach to automatic continuous vegetable oil refining by using unique process equipment, the design of which is suitable for automatic control. Essentially the process consists of two centrifugal contactors: a) a phase separator, referred to as Duozon, which separates refined oil from gum or soap stock; b) an extractor known as Hydrazon designed for water extraction of dissolved soap from the primary refined oil.

These machines have been described in a recent paper (1). The Duozon combines a large coalescing surface and high retention time with centrifugal force. The gravities however are not excessive enough to cause meal to hang up in the rotor; rather it is discharged continuously with gum or soap stock. Because of pressure operation and the high through-put capacity, a single unit may be used for primary gum or soap stock operation in refineries of two to 10 tank cars per day capacity. Gum or soap discharges from a single pipeline.

Similarly a single unit is used for multistage water-

---

[1] Present address: Chicago Refining Corporation, Chicago, Ill.