It is apparent that a reasonable estimate of the rate of bodying of many oils can now be arrived at from a consideration of the composition. Analysis of the behavior of mixtures of conjugated and non-conjugated oils should be carried out and correlated with the composition of the oils and their mixtures.

Liberation of Fatty Acids. When oils are bodied at temperatures of 300°C. and above for any prolonged time, the acid value shows a pronounced increase. If, as is occasionally done in the laboratory, bodying is carried out under vacuum, the liberated acids are largely removed with the volatile products. Attempts at continuous bodying at quite high temperatures have not been too successful, partly because of the high acidity of the bodied oils formed. The fact that liberated acids do not re-esterify suggests the possible formation of diglycerol esters.

Utilization of Bodied Oils

Heat-bodied oils are often used in paints and varnishes and are added to varnishes to check the bodying reaction. Bodied oils have been found to have considerably better durability than unbodied oils on outdoor exposure. To effect drying of an oil a certain amount of oxygen must first combine. The groups introduced by addition of oxygen in the drying-oil structure are focal points for the degradation reaction. The use of heat-bodied oils reduces the amount of oxygen necessary to effect the drying of the film and thereby improves its stability.

It is interesting to note that the dimer acids, formed by the heat treatment of fat acids, can be combined with the drying oils as are other dibasic acids in the formation of alkyd resins. These products have many properties of alkyd resins and indeed they should, but they also have much in common with bodied oils.

Heat bodying of oils is often carried out in the presence of natural or synthetic resins. Often the temperature required to disperse the resin is in the range usually employed in heat bodying. Thus some of the harder, less soluble resins cannot be used with tung or oiticica oils by conventional methods since the oils would gel before the resin was dispersed.

Since it is often undesirable to have unbodied oil present in a varnish, bodied oils are added to the varnish to chill it when the desired viscosity has been obtained.

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Blowing Drying Oils

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BLOWING with air or oxygen is a process applicable to all types of oils and fats. Hilditch states (11), "In many cases, especially with palm oils and tallows, it is possible to affect considerable bleaching by agitation with a current of air at 90-120°C. in enamelled, tinned, aluminum, or plain iron vessels." Blown whale and fish oils are used in stuffing greases for leather. Blown rapeseed oil and blown cottonseed oil are used in lubricants. The demand for blown unsaturated oils of the type with which we are concerned is evidenced by the fact that the Drying Oils Index of 1949, published by the Scientific Section of the National Paint, Varnish, and Lacquer Association (24), lists some 128 brand names of blown linseed, soybean, and fish oils. In addition, blown oils in considerable amounts are sold under brand names which are not listed or are prepared by various industries for use in their own plants. Also, blowing is recognized both in this country and abroad as a standard procedure in the preparation of the "boiled" oil used by house painters. Linoleum, which is the subject of a separate paper, has provided an outlet for fatty oils, mostly air oxidized, in such quantities that the Bureau of the Census lists the linoleum industry as one of the principal consumers of technical oils.

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Equipment

When commenting on the methods of conjugation, Cowan (6) states that blowing with air or oxygen is probably the oldest and is effective in promoting both conjugation and polymerization. Under the circumstances it is only natural that many devices are described in the literature for speeding up oxidation. Large oil surfaces may be exposed to a current of air. The air, or the oil, may be warmed. The oil may be agitated mechanically while air is blown through it, but if sufficient air pressure is maintained, no mechanical agitation is needed. On the other hand, the oil may be blown as a spray into a tank and recirculated until the desired degree of oxidation has been obtained. The design of the blowing equipment is important because it may affect the characteristics of the finished blown oil. The lack of agreement in the results reported in the literature may frequently be traced to differences in blowing equipment.

From the standpoint of economy it is often desirable to process large quantities of oil. In 1923 Anderson (1) recommended that for reasons of safety the size of a batch be limited to 25 barrels, but that quantity has been more than doubled and trebled whenever demand justified greater equipment capacity. The oil may be blown in large pans or horizontal cylindrical tanks, but refiners of drying oils have generally favored a vertical cylindrical tank. The ratio between the height and diameter of the cylinder may be varied according to the preferred ratio between air volume and the quantity of oil to be blown but is limited by the head of oil against which the blower will be effective. There is a tendency to increase the diameter of the cylinder when greater capacity is desired. The cylinder shell is usually extended at least five or six feet above the maximum level to which the tank will be filled in order to take care of turbulence and foam. The tank is covered and vented to a stack or condenser. A coil or jacket, or combination of the two, is provided for heating the oil with steam or cooling it with water. Instrument controls include thermometer, pressure gauges, and flowmeter for measuring air volume. Positive rotary or multistage centrifugal blowers supply air, which is released at the bottom of the tank. The speed of the oxidation reaction will be related directly to the extent of the oil surfaces exposed to the action of the air. Therefore various devices have been used to obtain the desired division of the air stream and at the same time provide agitation. The air may be released through perforated pipes (5) arranged in a coil or concentrically (13) or through a header (21) into smaller pipes arranged in star form (13) with end openings or perforations.

Laboratory Controls

Physical tests have been much favored for controlling the blowing process through examination of samples taken at intervals. Specific gravity determinations are practical and may be made quickly at the lower viscosities. Refractive indices furnish a useful means of control because of their accuracy and the speed with which they may be determined. Varnish makers are inclined to depend upon viscosity tubes, but direct reading viscosimeters have been found convenient. In fact, such viscosimeters and flowing sample visual refractometers may be included among instrument controls. Conclusions in regard to the oxidation and polymerization reactions occurring during the process may be drawn from molecular weight determinations and, in addition, O'Hare (28) recommends the determination of dielectric constants and power factors.

The chemical changes which take place during the course of the process will be evident from acid, saponification, and iodine numbers determined in the usual manner. The A.S.T.M. acid number method is generally used because of the effectiveness of the alcohol-benzene solvent mixture, but a method using even better solvents is desirable, especially in the case of highly oxidized samples. Determinations of petroleum ether insolubles provide some information about the increase in oxidized fatty acids, but much the same information is obtained in a practical way by determining the solubility in, or tolerance for mineral spirits (8). Further information can be obtained by determining hydroxyl and peroxide numbers, epoxy numbers, amount of conjugation, and ultimate analysis.

The Blowing Process

To quote Lundberg (20), "one of the best known facts about the autoxidation of olefins is that a host of secondary products may be formed. The kinds and quantities of such products vary greatly with the type of olefin oxidized, the extent of oxidation, oxygen concentration, temperature, presence or absence of positive and negative catalysts, and other factors." Because of this situation, the blowing process is frequently described in the most general terms. For instance, Mattiello (21) wrote, "the truly oxidized oils are blown to various extents, some of them to very heavy body. Where light color is desired and alkali refined oil is used as the starting oil. Where color is not so important, raw oil is used with the break left in. Temperatures are run to 220°F., then allowed to drop back to say 170°F. and the blowing continued; a header in the bottom with many small holes in it is used. The blowing is often continued until bodies as high as Z-2 are reached, each customer having a special degree of body and sometimes a solubility test." Although no exact or rigid formula can be given, it will be worthwhile to consider the usual stages in the blowing process and the facts involved when blowing the type of oil which Mattiello thought of as "truly oxidized."

Air drawn from within (7) or without the plant is probably the most common industrial reagent for oxidizing oils. Since hundreds of cubic feet of air may be used per minute, special treatment of this reagent would entail extra expense for equipment or operation. Oxygen has been used for experimental work. If air is used, it may be heated, dried, or purified. Ozonized air is liable to cause increased acidity. It is not ordinary plant practice, however, to subject the air to any other treatment than filtration to remove dirt or soot. The moisture content of such air varies according to the prevailing humidity. Novak (25) prefers to use humid air in making a conjugated type of blown oil, but ordinarily no special equipment is provided for regulation of humidity. It is emphasized, however, that oxidation will be speeded up if the air is forced into the body of oil in finely divided condition (13).

As stated, the oil selected for blowing may be a raw oil with the break left in. Blowing disperses the break thoroughly, and it tends to remain in suspension as the viscosity of the oil increases. Again, a well settled or mechanically clarified oil or a washed oil may be selected. A bleached oil may be selected in order to insure better color in the finished product. Other things equal, the acid value of the finished product will be lower if a neutral oil is used.

Induction Period

When air is first injected into a batch of oil, no easily detected reaction may occur for some time. Long (19) states that blowing may be conducted at low temperatures for many hours without apparent result. This induction period lasting for hours or days can be ascribed for the most part to inhibiting antioxidants present in natural oils. Removal of inhibitors by refining will speed up the start of oxidation, but it is claimed (23) that the reaction is autocatalytic and that the induction period cannot be eliminated entirely. Nevertheless, for practical purposes, the length of the induction period will be found to be related directly to the nature and amounts of the inhibitors present. The customary procedure for shortening the induction period is to turn steam into the heating coil or jacket and to blow the oil at a relatively high temperature until vigorous oxidation has started. Various starting temperatures have been mentioned in the literature. In one of his patents Novak (25) recommends 65-70°C. for low temperature processing. According to Lewkowitsch (17), the temperature is brought up to 70°C. or more, in some cases even to 110°-115°C. Mattiello (21) and Long (19) found that batches of linseed oil were heated to about 100°C. at the beginning of the blowing process. Hilditch (11) warns against temperatures above 120°C. when a pale oil of reasonably low acid value is desired, but temperatures up to 140°-150°C. for a short period are not unusual. Although such temperatures at the start of the process shorten the induction period radically, they are not high enough to cause any significant heat polymerization of unoxidized oil. Even when processing low temperature blown oil, Novak (25) concludes that a starting temperature of 70°C. may be used safely.

Light Blown Stage

At the end of the induction period some rather abrupt changes take place in the oil. First, there is some splitting of glycerides. The released glycerine is quickly oxidized to acrolein with its characteristic sharp odor. Second, the natural coloring materials in the oil are bleached by oxidation and heat. Good quality oils may have as little color at the maximum bleach point as Gardner Color Standards No. 3 or No. 2. Third, heat is generated as oxygen reacts with the oil. The reaction is exothermic and may require close control, particularly in the case of highly unsaturated oils. As soon as a change in color or odor of the batch are noted, the steam used to bring the batch up to starting temperature is reduced and then shut off. When the temperature continues to rise and threatens to exceed the prescribed starting temperature, cooling water is turned into the jacket or coil of the blowing tank. In fact, it is customary to cool the batch to a specified bodying temperature. In the cases previously cited Novak recommends reducing the temperature to 40°-45°C. Both Mattiello and Long found that it is usual practice to blow linseed oil at about 76°C. Chatfield (5) cites a range of 70°-82°C. for one typical process.

During any period of temperature adjustment the batch is agitated and a sufficient volume of air is used to keep the development of color down to a minimum. As the blowing is continued, increases in molecular complexity are reflected by higher specific gravities, refractive indexes, viscosities, and molecular weights. The process may be stopped at the light blown stage in the specific gravity range of .940 to .950 or a viscosity of about 1 poise; that is, the batch may be cooled, the air shut off, and the oil drummed out as aged oil, pale blown oil, patent leather oil, lithographic or ink oil. As these names indicate, the light blown oils can be expected to have improved drying characteristics, they may be finished with a light color; and they can be heat bodied rapidly in the varnish kettle to consistencies useful in the manufacture of inks or patent leather daub coats.

Heavy Bodied Stage

On the other hand, the blowing process may be continued to produce heavy bodied types. As the viscosity of the oil increases, there will be a gradual decrease in the vigor of the oxidation reaction and the process can be easily controlled even when the air volume is increased. Less and less heat will be generated, and a point will be reached where the temperature will start falling unless steam is again turned into the heating coil. Blown oils continue to polymerize at ordinary temperatures not only while being blown but also after they have been finished and packaged. Partly for this reason heavy bodied blown linseed oils are frequently finished in the Z-1 to Z-2 viscosity range. By the time they have reached the consumer, they are likely to be in the Z-2 to Z-3 range. Fish oils and semi-drying oils, that is, oils containing higher percentages of linoleic and less unsaturated fatty acids, are more frequently blown to viscosities of Z-4, Z-6 or higher. However the blowing of any of these oils may be continued as desired up to the point where a sample gels on cooling, when thinners such as mineral spirits or alcohols in amounts ranging from 10 to 30% are usually added.

The following tables are given as illustrations of the physical and chemical changes which occur in oils during the course of blowing operations.

TABLE I

Light Blown Linseed Oil
Starting Oil—Raw Linseed Oil

Blowing Time, Minutes	Temperature °C.	Specific Gravity 15.5/15.5°C.	Refractive Index at 25°C.	Acid Number	Color (Gardner)
0	25	.9334	1.47925	2.2	12
60	102	.9335	1.47927	2.2	12
90	137	.9338	1.47970	2.3	11
120	138	.9384	1.48017	2.5	10
140	138	.9404	1.48071	2.8	9
160	130	.9450	1.48120	3.0	9
180	108	.9497	1.48148	3.1	9
200	60	.9502	1.48152	3.3	9

Table I shows linseed oil blown and heated to a relatively high temperature and reacting with corresponding speed. By the time the oil had been heated to 137°C., it had passed through the induction period and thereafter the specific gravities and refractive indices increased steadily. After 60 minutes of blowing the oil had already started to bleach and in the interval between 120 and 140 minutes had been bleached to its highest color. The color change was more pro-

TABLE II

Heavy Bodied Blown Linseed Oil
Starting Oil—Raw Linseed Oil

Blowing Time, Hours	Temperature °C.	Refractive Index at 25°C.	Acid Number	Color (Gardner)	Viscosity Poises	Solubility in Mineral Spirits
0	10	1.4787	2.1	12	0.5	......
1	95	....	2.1	12	0.5	......
2	114	1.4789	2.2	11	0.6	......
4	121	1.4803	2.8	7	1.5	......
8	77	1.4844	4.2	7	......	......
17	71	1.4849	4.3	7	4.8	13.5
24	82	1.4854	4.7	....	......	8.7
32	79	1.4859	5.0	7-8	12.2	7.7
40	74	1.4865	5.3	8	18.8	5.6
45	48	1.4869	5.5	8	30.6	5.2

TABLE III

Heavy Bodied Blown Soybean Oil
Starting Oil—Degummed Soybean Oil

Blowing Time, Hours	Temperature °C.	Refractive Index at 25°C.	Acid Number	Color (Gardner)	Viscosity Poises at 25°C.	Solubility in Mineral Spirits	Peroxide Number	Hydroxyl Number
0	20	1.4713	.91	11	0.5	......	3.6	0.0
4	140	1.4722	1.1	5	0.65	......	78.3	4.1
6	71	1.4730	3.1	3-4	0.80	......	169.2	26.7
22	69	1.4750	3.0	3	1.65	......	413.8	20.7
47	70	1.4763	4.1	3	3.70	......	420.7	35.1
94	71	1.4776	6.3	3-4	8.84	19.1	368.5	43.3
126	70	1.4782	7.5	5	15.25	13.8	287.2	48.5
147	72	1.4790	8.2	5	25.60	9.2	297.0	72.4
166	68	1.4791	8.5	5	29.40	8.3	290.8	61.1

nounced than the figures indicate. The green of chlorophyll disappeared, and the orange red color of blown oils began to appear. Since this batch of light blown oil was to be finished at a specific gravity of .950, it was necessary to turn water into the coils and start reducing the temperature as soon as the oil was well bleached. Efficient cooling and good control is needed to have a batch such as this cooled to a safe packaging temperature at a specific gravity of .950.

Table II records changes occurring when a batch of linseed oil is blown to a typical heavy body but at a more moderate rate. At the end of two hours the temperature had been raised to 114°C., and the oil was just starting to oxidize. Although the temperature was not raised above 121°C. the oil was well bleached at the end of four hours and definite increases in refractive index, acid number, and viscosity were noted. The original oil had been completely soluble in or, stated otherwise, had infinite tolerance for mineral spirits, but after 17 hours of blowing an insoluble fraction separated after the oil was diluted with 13.5 times its weight of mineral spirits. As the process continued, this insoluble fraction increased until at the end of 45 hours the oil would tolerate only 5.2 times its weight of mineral spirits. On the other hand, there is an increase in the amount of material soluble in alcohol as the oil is processed from its original condition to the heavy bodied stage.

The figures for the blowing of a semi-drying oil follow much the same course, but at a slower rate. Table III illustrates the shortening of the induction period which occurred as a batch of soybean oil was heated to 140°C.

At the end of four hours the oil was bleaching, and in 22 hours the color had been reduced to Gardner 3. Later there was a characteristic development of chromophore groups and the color increased to Gardner 5. In addition, Table III gives some data indicating the increase and consumption of peroxides. The increase in hydroxyl number is significant and can be related to the changing solubilities in mineral spirits and alcohol.

In another case a batch of the same type of soybean oil was blown and heated to 80°C. and the temperature maintained in the 75°-80°C. range throughout the process. In Figure 1 the dotted line shows the viscosity increase of this batch. Practically no change was noted in the first 120 hours. For comparison the increase in viscosity in the Table III batch is shown in a solid line. This line shows the effect of a high starting temperature and a lower temperature after the bleach point. The dotted line shows the effect of a relatively low starting temperature and a higher temperature after the bleach point. Apparently the final products do not differ greatly. At refractive indices of 1.4790 and 1.4792 the iodine numbers were 76.4 and 75.4 and the solubilities 8.28 and 7.68, respectively.

Low Temperature Blowing

Processes such as those outlined in Tables II and III have been called standard. Some polymerization will have taken place and in the case of linseed oil, even when blown practically to the gel stage, the iodine number will be of the order of 125 Wijs and the oxygen content about 18%. By processing at lower temperature the iodine number can be reduced below 110 and the oxygen content raised to 20% (19). Temperatures as high as 95°C. have been mentioned in connection with low temperature processing but, when maximum oxygen content is desired, it is best not to exceed 70°C. at any time and to keep the temperature down to 50°C. after the bleach point. As the oxygen content increases, there is a correspondingly increased solubility in alcohol and other organic solvents composed of three elements such as carbon, hydrogen, and oxygen and decreased solubility in hydrocarbons (19). At a viscosity of 2.5 to 5.0 poises low temperature blown drying and semi-drying oils are soluble in natural and synthetic resins (25). Linseed oil blown at low temperatures for 200 or 300 hours or until it forms a solid gel on standing is compatible with nitrocellulose and nitrocellulose solvents (19).

High Temperature Blowing

Although the primary effect of blowing is oxidation, many of the desirable characteristics of blown oils are due to the complexity of the molecules built up during the process, or in succeeding processes in

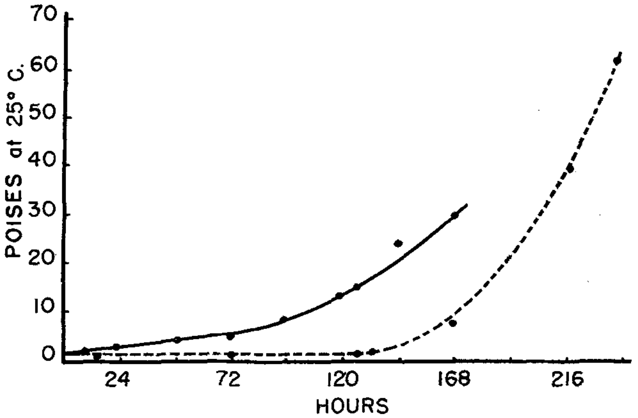

Fig. 1. Viscosity increases of two blown soybean oils.
Solid line ——— under conditions of Table III.
Dotted line – – – blown continuously in 75°-80°C. range

which blown oils are used. It has been observed that heat and the decomposition or consumption of peroxides formed during the process start the mechanism which promotes polymerization. For this reason, a number of processes have been developed in which much higher temperatures are used than any mentioned thus far. In the Asser (2) process, which was patented in Germany in 1929, linseed oil is heated to 280°C., and then air is "blown in while the heating of the material is continued to a temperature of 310°C. After about two hours an oil is obtained having a viscosity of normal stand oil." It is claimed that this type of oil has excellent drying and wetting properties, ready reactivity with resins, and some of the characteristics of tung oil (4). Considerable hydrolysis occurs during high temperature blowing and also cleavage at double bonds, resulting in the formation of short chain acids. If high acid values are undesirable, the process may be modified so as to promote recombination and re-esterification of fatty acids. In 1932 a patent was issued to Schwarcman (32) to cover a process in which linseed oil is cooked to 288°-316°C., cooled to 132°C., and then blown until the temperature is reduced to 99°C. and iodine number to 80-90. Long and McCarter (18) blew linseed oil at 110°C., 160°C., and 200°C. and obtained gels which contained 7.63%, 7.37%, and 6.98%, respectively, of oxygen, which might indicate that at the higher temperatures some heat bodying contributes to the gel formation and reduces the extent of oxidation required to lead to the same stage of molecular growth. Semi-drying oils have been blown in this temperature range and pilchard oil has been blown at 250°C. In the words of Lundberg (20), "evidence is now available that the mechanisms and products above 100°C. may be quite different from those at low temperatures."

Catalysts

Drier catalysts such as the salts of lead, manganese, and cobalt are used extensively in blown oil processes. Their action in accelerating the decomposition or consumption of peroxides and starting the mechanism which promotes polymerization resembles that of heat and, accordingly, oils blown with driers may be compared to those blown at high temperatures. Much of the boiled linseed oil produced for painters in this country and abroad is made by a process similar to that used in the production of light blown oil. According to Chatfield (5) boiled oils vary in specific gravity from .940 to .950 and are made by blowing oil at 90°-150°C. in the presence of soluble lead and manganese salts. According to Kirschenbauer (15) the oil is blown and heated. At about 100°C. the drier is added. Heating is discontinued and the exothermic action checked when the temperature reaches 125°-150° C. The term heavy bodied boiled when applied to an oil blown to a visvosity of Z-1 or higher signifies that it contains a drier or combination of driers. The characteristics of heavy bodied boiled oils can be varied somewhat by adding the drier at the start or at the end of the process.

As new protective coatings have been developed, blowing procedures have been examined and re-examined with a view to incorporating blown oils in new formulations. Blown oils have been developed for use with cellulose esters. They have been processed for use with natural resins. Blown oils of high viscosity react with phenolic resins (3) and Greaves (9) reports that this technique has been enormously developed. Low temperature blown oils have been used with alkyds and oil modified alkyds (25). In the development of styrenated oils the English have used polymerized oils and Americans have used blown oils (30). For some purposes the acids and hydrophilic groupings in blown oils are advantageous. When this is not the case, they may be reduced or removed (26) or recombined.

The development of blown oils for special uses has largely been the work of industrial chemists and production men. As late as 1949 Mills (21) wrote, "technical practice is still far ahead of theory, and much fundamental work remains to be done before the position can be reversed." Nevertheless the progress during the past decade in the chemistry of autoxidation and polymerization has been rapid and is providing a solid foundation for a study of the complex materials resulting from the combined action of both oxidation and polymerization. Blown oils will continue to interest both the chemist and the processor because they are active, highly functional materials.

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