This is chiefly a digest of sections of the following publications of the National Bureau of Standards dealing with linseed oil; its manufacture, types, and properties, and Federal Specifications covering these factors.

Circular C69, "Paint and Varnish", (November 17, 1917). 2


1 Specifications adopted by the Federal Specifications Executive Committee and approved by the Director of Procurement, Treasury Department, for use of all departments and establishments of the Government. Copies of all Federal Specifications mentioned in this digest may be obtained from Superintendent of Documents, Government Printing Office, Washington, D. C. (Price 5 cents).

2 Out of print and no longer available by purchase but may be consulted in Government depository libraries.

3 May be obtained free of charge from the National Bureau of Standards, Washington, D. C.

Manufacturing Processes

Linseed oil, the most important of the drying oils and the principal non-volatile paint vehicle, is obtained from flaxseed. It is produced commercially by grinding the seed, by the Anderson exceller mill process, and by extraction. Regardless of the process used, oil produced from the same seed has practically the same properties. However, there is a marked variation in oil from seed grown in different localities.

Grinding: The most common practice of producing commercial linseed oil is to grind the seed, heat the resultant meal with live steam to about 71°C or even as high as 32°C, and then subject it to hydraulic pressure.

Anderson Exceller Mill: In this process the ground meal is slightly heated and then forced by a screw against a conical grating through which the oil passes.

Extraction: In the extraction process the ground seed is treated with a light petroleum distillate, which extracts the oil, usually leaving less than 1/4 percent oil in the residue.

Types

Raw Linseed Oil is pure oil extracted from flaxseed. The oil as it comes from the press is cloudy, and although it is generally filtered and may appear to be clear, a certain amount of sediment settles after standing. This sediment called "foots" is considered objectionable. Perfectly clear oil is preferred for most purposes. The quality of raw linseed oil is more frequently influenced by the presence of excessive "foots" than by actual impurities. Paint made with oil containing considerable "foots" is liable to dry very slowly and may easily be washed off long after it is apparently dry. It is advantageous to filter all linseed oil before using, especially if it is cloudy.

The raw or unrefined oil when heated above 177°C frequently "breaks" and becomes cloudy. This is apparently due to the presence of non-oleaginous matter such as carbohydrates (mucilage), and phosphates of

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1Drying oil is "an oil which possesses to a marked degree the property of readily taking up oxygen from the air and changing to a relatively hard, tough, elastic substance when exposed in a thin film to the air"; "Standard Definitions of Terms Relating to Paint Specifications", American Society for Testing Materials, A.S.T.M. Standards (1933), pp. 735-739.

2"Foots" is a term applied to the solid or semi-solid matter that settles in the oil on standing. Its composition is quite variable, but it always contains some mucilaginous matter which is believed to hinder proper drying of the oil.

3"Break" is the thickening of linseed oil into clots, when it is heated.
calcium and magnesium. Although the mucilaginous substance causing the oil to break constitutes only a small fraction of 1 percent of the oil, such oil is not suitable for making varnish and certain other purposes. It is probable that this mucilaginous substance also forms at least a part of the "foots". Long storage tends to produce an oil from "foots", and one which will not break.

Refined Oil: Linseed oil is refined to remove all mucilaginous substances. Refining also removes some of the coloring matter and causes the oil to become somewhat less viscous. Refined oil is sometimes used in making paint, but is more commonly used in the manufacture of varnish which requires an oil that will not break.

Acid Process: The most common method of refining linseed oil is to agitate it with about 1 percent strong sulphuric acid, draw off the acid after the oil has settled, and wash the oil with water. This process generally increases, to some extent, the free acid content of the oil.

Fullers' Earth Process: In this process the oil is treated with Fullers' earth¹ at a temperature of about 71°C and then filtered.

Boiled Oil is a raw oil that has been treated with compounds of lead and sometimes suitable compounds of other drying metals, to produce a product that will dry rapidly. Boiled linseed oil² is now made by mixing "crusher's drier"³ with the raw oil in the proportion of about 1 to 16, and heating the mix until the drier is completely incorporated in the oil. It is generally believed that better oil results if the heating temperature is raised above 170°C than if, as is frequently done, the oil is simply heated by steam coils.

Boiled oils made from rosin are called resinate boiled oils, whereas those made without rosin are called linoleate boiled oils. If properly made, without an excessive amount of rosin, resinate oil is no doubt probably as good as the linoleate oil. Bunghole boiled oil is simply raw oil to which a drier, usually consisting of a solution of lead and manganese resinate or linoleate in turpentine or light mineral oil, has been added without heating.

¹Fullers' earth, an impure hydrous aluminum silicate, is a soft non-plastic earthy substance.
²The term "boiled linseed oil" is strictly a misnomer, since linseed oil does not boil as water does; it undergoes decomposition when heated to a sufficiently high temperature.
³Crusher's drier is a very concentrated boiled oil containing 15 to 20 times as much lead and manganese as is required in boiled oil. This drier is also made from rosin, forming resinates of lead and manganese.
The Bureau knows of no case in which boiled oil is used in paint, where equally satisfactory results could not be obtained by using raw oil and suitable Japan drier.

Bodied oil is produced by either heating raw oil, prepared so that it will not break by long storage or refining, to a temperature of about 280°C; or by blowing air through it at a temperature of about 121°C. The specific gravity and viscosity of the oil are both materially increased by the heating process. Blown oils are said to dry harder than oils bodied by heat alone.

Properties

Drying: Linseed oil, a fatty oil, is characterized by the presence of 'unsaturated' fatty acid radicles containing at least two double bonds which provide the property of absorbing oxygen from the air, thereby increasing the specific gravity and changing the oil into a hard horny mass when exposed in thin films.

As raw linseed oil dries so slowly, it is necessary to use a drier with it. During the early period of drying (the first 10 days) the oil increases in weight, 15 to 20 percent maximum, then gradually decreases until at the end of 150 days the film has resumed its original weight. The dry film has a much higher specific gravity and consequently occupies less space than the original oil. Even while the film is gaining in weight, volatile products such as water, carbon dioxide, and organic acids are given off. On outdoor exposures in clear warm weather, linseed oil requires two to six days for drying.

Color: The color (coloring matter derived principally from seed shells) of linseed oil varies from a pale straw yellow to a dark umber. Seed grown in a warm climate generally produces a lighter colored oil than that grown in a cold climate.

Linseed oil paints, made with oil of ordinary color, when applied on outdoor exposures soon bleach and become as light as those made with the palest oil. Although light colored (bleached) oil is usually preferred for interior work, all linseed oil in paint applied on indoor surfaces tends to turn yellow and to darken.

1 Fatty oils are, in general, mixed glycerides of fatty acids (radicles of fatty acids and glycerine in combination).

2 Fatty acid is "the acid which is present in fats or oils in combination with glycerine. As the fat breaks down, some of this fatty acid is liberated and small quantities of it are usually present in the free state. Fatty acids are entering into the recent paint trend in that fatty acids of certain oils are basic raw materials for synthetic resinous compounds", Ray C. Martin, "American Paint Journal", March 3, 1936.
Iodine Number,\(^1\) as well as the presence of excessive "foots", has considerable bearing on the quality of linseed oil. A very large proportion of linseed oil produced in the United States is crushed from Argentine seed which is generally characterized by a lower iodine number than that produced from North American seed. During the past ten years this difference has apparently been reversed, probably due to an increased planting of Bisom flax, adverse weather conditions or inadequate cleaning of the seed before crushing.

Many paint technologists believe that oil of a high iodine number is a better drying oil than that of a low iodine number. The only evidence to indicate that one is superior to the other is that the use of linseed oil of a low iodine number is likely to result in a paint which dries slowly, producing a soft film subject to a serious injury from dampness. Unless there is a material difference in price, it is advisable to purchase oil made from North American seed.

Federal Specifications

For Federal Specifications see JJJ-0-336 "Oil; Linseed, Raw\(^2\) covering two types, normal iodine number (177 minimum) and high iodine number (188 minimum), of the grade of raw linseed oil for use in paint; and JJJ-0-331 "Oil; Linseed, Boiled\(^2\) covering two types, kettle-boiled (0.937 minimum specific gravity) and quick process boiled (0.931 minimum specific gravity), of the grade of boiled linseed oil for use in paints.

Federal Specification JJJ-0-336\(^2\) limits the presence of "foots", by volume, to a maximum of 1 percent for heated oil, and 4 percent for chilled oil. As "foots" gradually settle, it is possible to draw off clear oil meeting requirements of the specification.

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\(^1\)Iodine Number: "Oils, waxes and resins have the property of absorbing iodine. It has been found that the percentage of iodine absorbed is fairly definite for each type of material and this iodine number is used as a basis of specifying and identifying products of a given quality. It also serves to indicate the capacity of drying oils to absorb oxygen from the air and, in conjunction with other tests, to determine the suitability of such oils for use in paint and varnish products", Ray C. Martin, "American Paint Journal", July 6, 1936.

\(^2\)May be obtained from the Superintendent of Documents, Washington, D. C. (Price 5 cents)