

UNITED STATES PATENT OFFICE.

WALTER D. FIELD, OF BLOOMFIELD, NEW JERSEY, ASSIGNOR TO THE
TANNETTE MANUFACTURING COMPANY, OF SAME PLACE.

MANUFACTURE OF OILS.

SPECIFICATION forming part of Letters Patent No. 574,408, dated January 5, 1897.

Application filed June 14, 1894. Serial No. 514,498. (No specimens.)

To all whom it may concern:

Be it known that I, WALTER D. FIELD, a citizen of the United States, residing at Bloomfield, in the county of Essex and State of New Jersey, have invented certain new and useful Improvements in the Manufacture of Oils; and I do hereby declare the following to be a full, clear, and exact description of the invention, such as will enable others skilled in the art to which it appertains to make and use the same.

My invention relates to that class of compounds that are used alone or in combination with other elements as a coating for textile fabrics, paper, or leather, and for insulating purposes and for various other purposes too numerous to mention.

The object of the invention is to so treat certain of the oils as to render them stable and durable, not liable to decompose or oxidize, practically odorless, and combinable with all kinds of pigments and other coloring-matters for coloring purposes.

It has long been recognized in the arts that combinations of different oils with other substances would be valuable, especially in making coatings for fabrics and for insulating and other purposes, the oils imparting to the compounds in which they are used a considerable degree of flexibility and also certain waterproofing qualities. These facts have led to many endeavors to use compounds containing oils for these purposes; but for many purposes there has always been a serious objection to using oils as one of the elements of the compound, and that objection has been the unstable character of most of the oils, and especially of those which, on account of their cheapness or other desirable qualities, it was most desirable to use, and by "instability" I mean the liability of these oils to decompose or become rancid and otherwise offensive under the conditions of their use. This objection of the instability of the oils has led to many efforts to discover some process of rendering these oils stable and not liable to decompose and become rancid, and many patents have been taken out, both in this country and in other countries, for such processes. Among other means for preventing such decomposition the combination of sulfur with

the oil to be rendered stable has been suggested, and patents have been taken out for different processes of combining sulfur with the oils in such a way as to render them stable, though many and probably most of these patents have related to the vulcanization of oils, either by sulfur or chlorid of sulfur, in such a way and to such a degree as to materially change the physical characteristics of the oil and make a tough or hard and practically insoluble body, thereby destroying its usefulness for many purposes where a readily soluble body is necessary.

I myself have taken out a patent in this country for combining sulfur with the oil to be rendered stable by the use of chlorid of sulfur, which patent is numbered 498,162 and is dated May 23, 1893, and also for combining sulfur with the oil to be treated by the use of sulfuric acid, which patent is numbered 478,955 and dated July 12, 1892, and I have also taken out a patent in this country for the combination of oils after treatment with sulfur with pyroxylin, which patent is numbered 473,306 and is dated April 19, 1892, in which patent I describe the method of making the well-known substance sulfur balsam. I have also taken out a patent in this country for the combination of an oil rendered stable by the action of air, known as a "blown" oil, with pyroxylin, which patent is numbered 491,880 and is dated February 14, 1893. I cite these patents as illustrations of the efforts that I have made to discover some satisfactory process of rendering the oil stable, and also of showing the combinations or compositions in which the oil so rendered stable may be used.

I have found in practical use that the objection to the oils rendered stable by the combination of sulfur when combined with the oil in any of the ways heretofore proposed, whether by the use of chlorid of sulfur, sulfuric acid, or by the introduction of the sulfur directly, as in making sulfur balsam, is twofold: First. When the sulfur is introduced in any of these ways, either some free sulfur remains in the oil or, as more frequently happens, a considerable amount of sulfureted hydrogen remains dissolved in the oil, which it is impossible to practically get rid of, or

various polysulfids are formed, which also remain in the oil. These different forms of sulfur all impart to the oil a very strong and offensive sulfur odor, which practically renders it of no value in compounds otherwise adapted for indoor uses. Second. This free sulfur, sulfureted hydrogen, and these polysulfids remaining in the oil attack and destroy many of the pigments and other coloring-matters which are desirable to use in compounds of which this oil forms an element, thus very much limiting the pigments that can be used and the range of colors that can be obtained in compounds containing these sulfureted oils.

I have also found that while the oil acted upon by oxygen, known as "blown" oil and described in my Patent No. 491,880, is free from the objection of containing free sulfur and these other sulfur compounds, it is open to the objection that it is very difficult, when operating on a manufacturing scale, to render the oil perfectly stable by this process, and some parts of the oil probably remaining unacted upon or imperfectly acted upon by the oxygen contained in the air the oil when used in combination with other elements in the arts tends to decompose and become rancid and offensive in odor, and when used in certain combinations, as a coating for textile and other fabrics, it imparts a softness to the coating which is undesirable and prevents the use of the coated fabric for many purposes.

I have therefore sought some method of rendering the oil to be used stable and not liable to decompose and become rancid which would overcome these objections and would combine the good feature of the sulfureted oil—viz., its stability—and the odorless quality of the blown oil without the disadvantage of either, and I have succeeded to the extent hereinafter set forth.

I have discovered that if during the process of blowing oils described in my Patent No. 491,880 a sulfid of any one of the group of metals known as "pentavalent" metals be added to the oil being blown in proper proportions and under proper conditions the sulfid will be taken up into chemical union or combination with the oil and will greatly increase its stability. I have availed myself of this fact in my hereinafter-described process.

The oils to which this invention relates and which are to be treated by my process are known in the trade as the "non-drying" oils and include all of the group of oils technically known as the "glycyl-ethers of the unsaturated fatty acids" except the "linseed-oil" group, though some of these oils which contain considerable proportions of the glycerids of the saturated fatty acids, such, for instance, as palm-oil, are not as easily treated as the others and not as useful for many purposes in the arts after treatment. I have most commonly treated cotton-seed oil, olive-oil, and maize or corn oil, and have for my purposes secured the best results from the

use of these oils, although any of the other oils heretofore described may be used successfully, and for some purposes some of them are preferable to the ones last named.

I shall use in describing my process the oil commonly known as "cotton-seed" oil.

I take one hundred pounds of cotton-seed oil and place it in a vessel which I use for preparing blown oils, and which may be any vessel ordinarily used for such purposes, and requires no description here. I then raise the temperature of the oil to about 200° centigrade, preferably with constant blowing with air, and when that degree of heat is reached I add to the one hundred pounds of oil one-half of one pound of penta sulfid of antimony, which penta sulfid is commonly known as the "golden sulfid of antimony." I then continue to pass air through the oil or blow the oil with air, keeping the temperature at about 200° centigrade until the oil reaches the proper density, and a practical test to determine when this point is reached is that the oil will string between the thumb and forefinger, when outstretched, without breaking. When that condition of the oil is reached, I permit the oil to cool, and it is then ready for combination with other substances for use in the arts. It will be found that during this treatment of the oil no sulfureted hydrogen is given off, thus showing that there is no free sulfur in the compound, and after the treatment is complete it will be found that no free sulfur or any of the undesirable sulfur compounds remain in the oil, but that the sulfid has been entirely taken up into chemical combination with the oil. These facts are shown by the fact that the oil so treated can be used with the "anilin lakes," colors which are destroyed by the presence of free sulfur, and also by the fact that the oil does not respond to any of the tests for sulfur commonly used. It will also be found that the oil is practically odorless and can be combined with any pigments or coloring-matters desired. At the same time the oil so treated is fully as stable as the oil treated with pure sulfur and much more stable than the oil treated with air alone.

One of the advantages of this process is that the progress of oxidation by air when the penta sulfid of antimony is added to the oil is very much slower than when the penta sulfid is absent, and the oxidation can be carried further and be made more complete without the danger of carrying it too far and solidifying the oil which exists when oil is blown without the sulfid of antimony, and this feature is of peculiar advantage when operating with these oils on a manufacturing scale. This oil so treated with sulfid of antimony and air is soluble to any extent in petroleum naphtha and the usual oil solvents. It can be combined with pigments and pyroxylin and used in the arts as a coating for textile fabrics, paper, and leather, or it can be combined with other suitable substances

to give it body and be used as an insulating material; but I do not claim any of these herein.

It is proper to add that I have only used the penta sulfid of antimony as illustrating my process, and the other sulfids of antimony and the sulfids of any of the pentavalent metals, such as the sulfate of arsenic and bismuth, for example, may be used instead of the penta sulfid of antimony, and also that while I have found the proportions of one-half pound of sulfid of antimony to one hundred pounds of oil to be successful in my use of them, the sulfid of antimony and the other sulfids may be used in considerably larger proportions under proper conditions, only care should be taken not to use larger proportions of the sulfids than will completely combine with the oil. It is also proper to add that while I have described an oil raised to a temperature of about 200° centigrade in illustrating my invention, I do not intend to limit this invention to that temperature. I have found that temperature to be the best in the practical working of this invention, but it is true that the same results can be reached by using a temperature as low as 150° centigrade or as high as 210° centigrade, (which I have found to be practically the limits,) but it will be found that when temperatures lower than 200° centigrade are used the operation will be proportionately slower, and where higher temperatures than 200° centigrade are used that there is danger of a too rapid action, resulting in a possible solidification of the oil, and there is also danger of decomposing the oil.

Having thus described my invention, what

I claim, and desire to secure by Letters Patent, is—

1. The herein-described process of rendering stable the glyceryl ethers of the unsaturated fatty acids, which consists in raising them to the temperature of from 150° to 210° centigrade then adding the sulfid of a pentavalent metal and then injecting air into the mixture until it reaches the required density.

2. The herein-described process of rendering stable the glyceryl ethers of the unsaturated fatty acids, which consists in raising them to the temperature of from 150° to 210° centigrade then adding sulfid of antimony and then injecting air into the mixture until it reaches the required density.

3. The product resulting from the process of rendering stable the glyceryl ethers of the unsaturated fatty acids, which consists in raising the temperature of the glyceryl ether from 150° to 210° centigrade and then adding the sulfid of a pentavalent metal and then injecting air into the mixture until it reaches the required density.

4. The product resulting from the process of rendering stable the glyceryl ethers of the unsaturated fatty acids, which consists in raising the temperature of the glyceryl ether from 150° to 210° centigrade then adding a sulfid of antimony and then injecting air into the mixture until it reaches the required density.

In testimony whereof I affix my signature in presence of two witnesses.

WALTER D. FIELD.

Witnesses:

HARRY E. RICHARDS,
J. D. GALLAGHER.