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PROCESS FOR REMOVING MARKS ON WOOL

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Our invention relates to a method for the treatment of wool in order to remove therefrom the marks which have been applied thereto for the purpose of distinguishing its origin. As is well known, such identifying marks, made with tar oil, are generally applied to the wool on the sheep. The tar oils constituting these marks afterwards lose their volatile components and become hard under the action of the oxygen of the air. Such marks usually contain tar or other coloring substances which soil the wool. The object of this invention is to remove these marks, and this is accomplished by a treatment which renders them again liquid.

Heretofore it has been generally common for many years to wash and scour the wool for removing the accumulated dirt, to remove its natural grease, and also, to the extent possible by these prior methods, with the object of removing any identifying marks of coloring matter which may have been applied thereto. In this prior practice, in some instances various tar oils, as well as petroleum products, have been employed. It is well known that this prior practice has not been entirely satisfactory and that it has proved more or less ineffective for thoroughly removing the identification marks above noted.

In carrying out the present invention, the wool may have been previously subjected to such a cleansing treatment for the removal of the natural oil or grease, etc., particularly in cases where the original wool is more than ordinarily greasy. On the other hand, the present invention may be carried out in a combined operation for degreasing the wool and at the same time removing the marks therefrom in a single complete treatment, particularly in case the original natural wool contains only a relatively small amount of grease. In this latter case, the heavy tar oil employed in carrying out the process of this invention, will dissolve and remove the grease.

According to this invention, as hereinafter particularly set forth, special oils and solvents are employed together with a particular process or treatment by which it has been found that the identification marks are effectively removed from the wool. Such process further includes substantially the complete recovery of the materials used, which are then again reused in continuing the carrying out of the process or method.

In carrying out the method of our invention, the wool is first thoroughly soaked at a temperature within the limits between 30° C. and 70° C., in a digester containing heavy tar oils which are free from anthracene and also free from any substances the boiling point of which is lower than 100° C. After the removal from the wool of the greater portion of these oils, the remainder still contained in the wool may be removed by means of benzine and petroleum ether, or other suitable volatile solvents, as the remainder of the heavy tar oil still contained in the wool will dissolve in this mixture and thus can be extracted from the wool. The mixture thus obtained of benzine, petroleum ether and heavy tar oil is then subjected to fractional distillation, thereby to recover the benzine and the petroleum ether. The tar oil, which contains portions of the marks and other coloring substances, may be separated from these substances. These coloring substances, which are contained in the tar oil, comprise both soluble and insoluble substances. The insoluble substances are separated by decantation, and the solution which remains contains the tar oil, the dissolved portions of the marks and the soluble coloring substances. For enabling reuse of the tar oil, this solution may be put through a fractional distillation, whereby the different soluble substances are separated, while the insoluble coloring substances remain in the distillation apparatus.

When the wool is put into a digester containing the heavy tar oil, the wool may or may not have been previously degreased. The heavy tar oil, chosen, as above noted, to have a boiling point higher than 100° C., may comprise naphthalene oil of the ortho position 1, 2, such oils being obtained from naphthalene by fractional distillation and subsequent cooling.

When the marks have been dissolved by soaking the wool in a tar oil of the kind above noted, the liquid contents of the digester are emptied out and the wool in the digester

which still contains a substantial amount of the tar oil, is then cleaned. To effect this cleaning the wool is treated, in a substantially usual way, by means of a volatile solvent of the kind commonly used for degreasing the wool, such, for example, as benzine, or petroleum ether. When the wool has been thus cleaned and thereby freed from the remaining tar oil which it still contained after the first treatment, the liquid contents of the digester are again emptied and steam approximately at a temperature of 100° C. is admitted into the digester. This steam removes the above noted solvent from the wool so that thus a completely clean wool free from marks is obtained.

In order to carry out the method in a manner which would prove economically workable, the materials used, such as the benzine, petroleum ether, and the tar oil, should be recovered. This recovery is effected by fractional distillation. By reason of the fact that the boiling point of the tar oil is higher than 100° C., the benzine and the petroleum ether may be readily separated out and recovered by distillation, leaving behind only the tar oil. The recovery of the tar oil from the mixture of dissolved tar and other coloring matter may be effected by further fractional distillation. For effectively carrying out the method, the tar oil used must be completely soluble in the volatile solvent used and must be incapable of forming a soap in the presence of water. In some instances it may be desirable that the tar oils should have mixed therewith light coal oil derivatives, such as the solvent generally known as "naphtha solvent," or a heavy benzol. If desired, in the recovery of the tar oil, the insoluble substances, such as the carbon, may be separated by decantation, while the soluble substances are separated by a fractional distillation.

An example of a particular manner in which the process of this invention may be carried out is as follows:

The wool to be treated is introduced into a digester which may then be closed. A heavy tar oil of the kind hereinbefore described is now run from an elevated tank or containing vessel through a pipe connection into the digester. The digester is filled with such oil to a sufficient extent that the wool is completely covered and submerged therein. The wool is now permitted to soak in this heavy tar oil for a sufficient length of time until the hardened material comprising the marks has become thoroughly softened and dissolved in the oil. The length of time this soaking should continue may be readily determined and closely estimated in practice depending upon the original condition of the wool; and the digester may be opened for the inspection of the condition of the wool at any time, and, in fact, may be left open for such purpose during this part of the process if desired. The duration of this treatment of the wool with the heavy tar oil may be from twenty minutes to one hour. Also, if desired, during the soaking operation, the tar oil used for that purpose may be permitted to flow slowly through the digester and wool from top to bottom. In any case, some of such tar oil may be drawn off at any time, for inspection, and more of it admitted into the digester.

After the material comprising the marks has been thus softened and dissolved, the liquid tar oil with such material dissolved therein is drawn off from the bottom of the digester, through a pipe connection, into a reservoir. After the digester has been thus emptied of its liquid contents, it should be understood that the wool in the digester still contains therein some of the tar oil which also must be removed. To accomplish this latter result, a volatile solvent, such as benzine, or petroleum ether, is introduced into the digester through a pipe connection from an elevated containing vessel or tank, until the digester is again filled sufficiently to cover and submerge the wool in this volatile solvent. From time to time this volatile solvent, having portions of the tar oil dissolved therein, is drawn off from the bottom of the digester, through a pipe connection, into a receiving vessel. This part of the operation is continued until the volatile solvent which is thus drawn off appears to be completely pure and free from dissolved substances.

It is now desirable to remove from the wool the volatile tar solvent, some of which remains therein. Also it is desirable that the wool should be thoroughly cleaned. For effecting these results, steam having a temperature of 100° C. is introduced into the closed digester at the bottom thereof through a pipe connection leading from any convenient source, such as a steam boiler. The top of the digester has a connection to a condenser through which the steam passes after it has passed through the digester and its contained wool.

This steam evaporates the volatile solvent and carries the latter out with it into the condenser, where both the steam and the volatile solvent carried thereby are condensed to a liquid. This liquid, comprising water and the liquid solvent, flows out of the condenser into a receiving vessel, in which they separate by reason of their different specific gravities. From this vessel the water may be drawn out and the volatile solvent is returned therefrom by means of a pump into its original tank or container above noted, for reuse. When no more of the volatile solvent, but only water, is recovered from the condenser, this part of the process will have been completed. However, experience and practice readily determine the length of time that the steam should be passed through the digester. The shutting off of the steam concludes the process in so far

as the wool is concerned, and the wool may now be removed from the digester. Such wool will be in a thoroughly clean condition, free from oils, grease and marks. As above noted, if the original wool contains a comparatively large amount of the natural grease, such wool will have been previously degreased, however, with wool originally containing only a relatively small quantity of grease, the preliminary degreasing operation may be omitted, and in such case, this grease may be removed, together with the marks, in carrying out this process, as so far described.

As a feature of the complete process, it is desired to purify and recover from its dissolved oils, etc., the volatile solvent which, as above described, was drawn from the digester into a separate vessel or container. For accomplishing this result, this mixture containing the volatile solvent is pumped from its container into the digester, whereupon the steam is again passed through the digester and then through the condenser, as above described. This steam evaporates the volatile solvent and carries the latter away with it and through the condenser. This part of the operation is continued until no trace of the condensed or liquid solvent can be found issuing from the condenser.

When this last part of the operation is concluded there will remain in the digester the heavy tar oil with its dissolved impurities, as above described, and also there will be some water in the digester which has been produced by condensation of the steam. Because of their different specific gravities, this tar oil and the water may be readily separated by decantation. The water may be first drawn off from the bottom of the digester and then the heavy tar oil may be drawn off, the latter being delivered into a retort.

Also the heavy tar oil with its dissolved impurities which was drawn off from the digester following the soaking operation in the first part of the treatment and was received in a containing vessel, is now pumped from this container into the same above noted retort. This heavy tar oil now in the retort contains therein the tar oil which was dissolved out of the wool and which formed the marks thereon. In case the wool had not been previously degreased, the dissolved grease is also contained in this heavy tar oil in the retort. Also it contains considerable dirt and other impurities. This mixture of the heavy tar oil in the retort is now subjected to fractional distillation. The still or distilling apparatus comprises, besides the above noted retort, a usual or suitable furnace, receiving chambers, etc.

In this fractional distillation the first fraction is the heavy tar oil in its purified form, which is collected in a receiver or reservoir. This first fraction, the heavy tar oil, is obtained at temperatures between 100° C. and 190° C. From this reservoir this distilled oil is pumped back into the hereinbefore noted tank from which it was first supplied to the digester, to be reused at a subsequent treatment of wool in the digester. The above noted grease forms the second fraction of this distillation and is collected in another receiver or reservoir, to be used for any purpose desired. By heating to a temperature of 250° C., all the olein of the wool is distilled and the residue is more or less hard tar. The solid matter and any heavier impurities of course remain in the retort.

It will now be evident that the process or method of the present invention provides for the recovery and reuse of all of the solvents which have been used. Also it will be understood that this process may be combined with the degreasing process, in the manner hereinbefore set forth. In some cases, however, in order to effect the degreasing, it may be advisable to make use of petroleum ether, such as, a mixture of benzine, petroleum ether and tar oil. In such case, the procedure may be substantially as follows:

A mixture of petroleum ether, naphtha solvent and rather light tar oil is prepared, the wool is soaked in this mixture, and may remain in it from twenty minutes to one hour. The mixture, which then contains the dissolved matter, is then evacuated. The wool is then washed with petroleum ether, to clean it. The solution is afterwards decanted, if the latter is to be used again for a similar new operation without its fractional distillation. When this solution is subjected to distillation, the petroleum ether is distilled at temperatures lower than 100° C., the naphtha solvent between 100° C. and 160° C., the tar oil between 160° C. and 200° C., and the grease between 200° C. and 250° C. The residue comprises regenerated tar which is too heavy for distillation.

However, regardless of the particular way in which the invention may be carried out, that is to say, whether the objectionable coloring marks are dissolved and removed simultaneously with the degreasing of the wool, by reason of the mixing of a volatile solvent with the tar oil, or whether the marks are removed in a subsequent treatment of the wool, which has been previously degreased, it should be evident that this invention provides economy in working by reason of the complete recovery for reuse of the various elements; and also it will be noted that there is no necessity for first passing the original greasy, dirty and marked wool through a crusher or the like for thereby more or less pulverizing the hard clots and lumps, which in part are formed from the hardened oil used in marking the wool on the sheep.

It is obvious that various modifications may be made in the process above particularly de-

scribed, within the principle and scope of the invention as defined in the appended claims.

What we claim is:

1. The method of removing marks from wool, which consists in soaking the wool in a digester in heavy tar oils the boiling point of which is higher than 100° C., in drawing off from the digester as much as possible of the said heavy tar oils, and in treating the remaining tar oils still contained in the wool with a mixture of volatile solvents whereby the said remaining tar oils are dissolved.

2. The method of removing marks from wool, which consists in soaking the wool in a digester in heavy tar oils the boiling point of which is higher than 100° C., in drawing off from the digester as much as possible of the said heavy tar oils, in treating the remaining tar oils still contained in the wool with a mixture of volatile solvents whereby the said remaining tar oils are dissolved, and in recovering the solvents from the mixture of volatile solvents and of heavy tar oils.

3. The method of removing marks from wool, which consists in soaking the wool in a digester in heavy tar oils the boiling point of which is higher than 100° C., in drawing off from the digester as much as possible of the said heavy tar oils, in treating the remaining tar oils still contained in the wool with a mixture of volatile solvents whereby the said remaining tar oils are dissolved, and in recovering the heavy tar oils from the heavy tar oils drawn off from the digester having therein dissolved and undissolved substances.

4. The method of removing marks from wool, which consists in soaking the wool in a digester in heavy tar oils the boiling point of which is higher than 100° C., in drawing off from the digester as much as possible of the said heavy tar oils, in treating the remaining tar oils still contained in the wool with a mixture of benzine and petroleum ether whereby the said remaining tar oils are dissolved, and in recovering the solvents from the resulting mixture of benzine and petroleum ether and dissolved heavy tar oils.

5. The method of removing marks from wool, which consists in soaking the wool in a digester in naphthalene oils, in drawing off from the digester as much as possible of the said naphthalene oils, in treating the remaining naphthalene oils still contained in the wool with a mixture of volatile solvents whereby the said remaining naphthalene oils are dissolved, and in recovering the solvents from the mixture of naphthalene oils and of volatile solvents.

6. The method of removing marks from wool, which consists in soaking the wool in a digester in heavy tar oils mixed with light coal oil derivatives, in drawing off from the digester as much as possible of the said mixture of heavy tar oils and light derivatives, and treating the remaining mixture of tar oils and light derivatives still contained in the wool with a mixture of volatile solvents thereof.

In testimony whereof we have affixed our signatures.

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