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COMPOSITION FOR TREATING LEATHER
AND PROCESS FOR PREPARING THE
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This invention relates to compositions for treating leather and to processes for preparing the same; more particularly this invention relates to compositions suitable for fatliquoring leather.

It is well known that sulfated oils such as sulfated cod oil, sulfated neat's-foot oil, sulfated sperm oil, sulfated castor oil, or sulfated rapeseed oil used for fatliquoring leather should have pH values of between about 5 and about 7.5, preferably between about 5 and about 6, depending upon the particular type of leather to be treated. Such oils ordinarily contain water in the amount of between 10% and 50% of the oil and may contain, in addition, raw oils such as natural or manufactured moellon, and varying amounts of salts and other impurities. Many of these sulfated oils tend to become cloudy at pH values suitable for fatliquoring leather, the clouding being chiefly due to separation of water, raw oil or other substances from the sulfated oil. Cloudy sulfated oils give poor results when used for fatliquoring leather as the oil does not penetrate the leather properly and a spotty product may thus result. While these cloudy oils may be cleared by treatment with alkalis such as sodium carbonate or ammonia, the pH value of the oil is raised to such an extent that when the clear oils are used for fatliquoring leather, the proper take-up thereof is not accomplished, the leather may become harsh, and if dyed leather is being treated, the color thereof may be substantially injured.

The object of this invention is to produce sulfated oils suitable for fatliquoring leather which are clear and substantially permanently stable at pH values between about 5 and about 7.5.

It has now been found that clear and substantially permanently stable sulfated oils having pH values between about 5 and about 7.5 may be prepared by incorporating in a sulfated oil having a pH value between about 5 and about 7.5, a relatively small amount of an organic nitrogen compound selected from the group consisting of amides and thiamides. The incorporation of these organic nitrogen compounds in such sulfated oils prevents clouding of the oils, or if clouding has already taken place, effectively clears the oils; such compounds do not, however, increase the pH of the oils to any substantial extent so that the oils are eminently suitable for fatliquoring leather and like purposes. For the sake of brevity, the term "organic nitrogen compound" hereinafter used is intended to mean an

organic nitrogen compound selected from the group above set forth.

The organic nitrogen compounds which may be employed in accordance with this invention may be mono-, di-, or poly amides, or thiamides; furthermore, secondary and tertiary as well as primary organic nitrogen compounds of the above types may be used. The following organic nitrogen compounds may be mentioned as specific examples suitable for use in the practice of this invention: urea, acetamide, di-acetamide, tri-acetamide, N-ethylacetamide, acetanilide, thiourea, thiacetamide, thiacetanilide. The particular organic nitrogen compound employed depends to some extent upon the sulfated oil to be treated. Thus, for example, urea has been found suitable for clearing a sulfated cod oil composition or a mixture of sulfated sperm oil and coconut oil containing water. Under certain conditions of operation it may be found that other organic nitrogen compounds may be more efficient than urea; it is intended that in practicing this invention, the combination of organic nitrogen compound and sulfated oil found to be most effective under the particular conditions of use should preferably be employed.

The compositions of this invention may be prepared by sulfating an oil such as cod oil in any suitable manner, washing the product with water, partially neutralizing the oil to a pH value between about 2.5 and about 5, preferably between about 2.5 and about 4, so that substantially all of the free sulfuric acid is neutralized, settling the partially neutralized oil in a pan in a warm room and drawing off the salt and acid water. The partially neutralized oil thus prepared may be further treated with an alkali such as soda or ammonia to adjust its pH value to between about 5 and about 7.5, preferably between about 5 and about 6. The oil may then be treated in accordance with this invention by adding sufficient amounts of an organic nitrogen compound to produce a clear, stable, sulfated oil product. The amount of the organic nitrogen compound added to the oil may vary to some extent, depending somewhat upon the particular oil being treated; it has been found that generally an amount of organic nitrogen compound equivalent to between about 0.5% and about 5% of the final product is suitable. Sufficient water may then be added, if necessary, to adjust the moisture content of the sulfated oil to between about 10% and about 50%, preferably between about 25% and about 30%, depending upon the particular type of product desired. Raw oils,

i. e., oils which have not been subjected to solubilizing treatments may be added to the oil if desired. If cloudiness has been induced by the addition of water or raw oils, additional amounts of an organic nitrogen compound may be added to the oil in order to produce the desired clarity; this, however, is ordinarily unnecessary. The finished product thus obtained is a brilliantly clear, stable, sulfated oil having a pH value of between about 5 and about 7.5, preferably between about 5 and about 6.

The above method of preparation may be varied somewhat. If desired, the oil may be neutralized to a pH value of between about 5 and about 7.5 before panning. The organic nitrogen compound may be added to the oil before neutralization or may be introduced into the oil after water, raw oils or both have been added. The organic nitrogen compound may be added to the oil in a substantially pure condition or in the form of a solution or suspension in a liquid medium compatible with the oil; thus, for example, when using urea as the organic nitrogen compound, it may be added to the oil in the form of an aqueous solution thereof. Other modifications may also be made without departing from the scope of this invention.

This invention is also applicable to the treatment of sulfated oils containing water, raw oils or both which have become cloudy on standing. It has been found that the addition of between about 0.5% and about 5% of an organic nitrogen compound to such cloudy oils effects an immediate clarification of the oils and produces products which remain substantially permanently stable for long periods of time.

In some instances, it may be desirable to incorporate a reducing agent such as sodium bisulfite in the oil along with the organic nitrogen compound, since it has been found the presence of such reducing agents tends to increase the stability of the cleared sulfated oils; generally, an amount of reducing agent equivalent to between about 0.25% and about 1% of the final product is suitable.

The following examples are illustrative of this invention. Amounts are given in parts by weight.

Example I

A composition comprising a mixture of sulfated sperm oil and coconut oil containing about 30% water and having a pH of about 5.8 became very cloudy and an appreciable amount of water separated after standing for a short time. About 3 parts of urea were added to about 97 parts of this composition. The mixture was then stirred and warmed gently. The composition cleared to form a brilliantly clear product, all the separated water being taken up again by the oil. Furthermore, the product remained clear after repeated freezings and thawings. The addition of the urea to the composition did not raise the pH value to any substantial extent.

Example II

A composition comprising about 58 parts of a sulfated cod oil, 19 parts of a natural moellon, 19 parts of a manufactured moellon made by blowing warm air through raw cod oil and containing about 25% water, and about 4 parts of urea was formed. The pH value was adjusted to 5.9 with soda; the composition thus obtained remained clear and stable. A composition containing the above ingredients in the same rela-

tive proportions but containing no urea clouded at a pH value of 5.9 in a short time.

Example III

A composition comprising about 78 parts of a mixture of sulfated sperm oil and coconut oil, 10 parts of a sulfated mixture of coconut fatty acids and castor oil, 9 parts of water and 3 parts of urea was formed. The pH value was adjusted to 5.7 with soda; the composition thus obtained remained clear and stable. A composition containing the above ingredients in the same relative proportions but containing no urea clouded at a pH value of 5.7 in a short time.

Example IV

A composition comprising 97 parts of a mixture of sulfated sperm oil and coconut oil containing about 30% water and 3 parts of acetamide was formed. The pH value was adjusted to 5.8 with soda; the composition thus obtained remained clear and stable. The mixture of sulfated sperm oil and coconut oil containing about 30% water but containing no acetamide clouded at a pH value of 5.8 in a short time.

Example V

A composition comprising 97 parts of a mixture of sulfated sperm oil and coconut oil containing about 30% water and 3 parts of acetanilide was formed. The pH value was adjusted to 5.8 with soda; the composition thus obtained remained clear and stable. The mixture of sulfated sperm oil and coconut oil containing about 30% water but containing no acetanilide clouded at a pH value of 5.8 in a short time.

Example VI

A composition comprising 97 parts of a mixture of sulfated sperm oil and coconut oil containing about 30% water and 3 parts of thiourea was formed. The pH value was adjusted to 5.8 with soda; the composition thus obtained remained clear and stable. The mixture of sulfated sperm oil and coconut oil containing about 30% water but containing no thiourea clouded at a pH value of 5.8 in a short time.

Example VII

A composition comprising 96.5 parts of a mixture of sulfated sperm oil and coconut oil containing about 30% water, 3 parts of urea and 0.5 part of sodium bisulfite was formed. The pH value was adjusted to 5.8 with soda; the composition thus obtained remained clear and stable for 10 months when it was used. A mixture of sulfated sperm oil and coconut oil containing about 30% water but containing no urea and sodium bisulfite clouded at a pH value of 5.8 in a short time.

The above examples clearly show that the addition of the organic nitrogen compounds to sulfated oils produces substantially permanently clear products having pH values within the range suitable for treating leather. The chemical action of these compounds on the sulfated oils is somewhat obscure due to the extremely complicated and varied character of the oils treated in accordance with this invention. Thus the oils before sulfation may contain degradation products such as di- or mono-glycerides and fatty acids as well as other substances of a more or less complicated nature. In addition, the sulfuric acid treatment ordinarily causes the formation of various hydrolytic decomposition prod-

ucts, thereby increasing the complexity of the chemical mixture being dealt with. The compounds which may be used in accordance with this invention may be either basic as in the case of urea, or neutral as in the case of acetamide; yet these compounds act in an entirely similar manner when added to sulfated oils in accordance with this invention. In view of the above, it will be seen it is impossible to advance any theory to explain the action of the organic nitrogen compounds used; in some cases the organic nitrogen compound may react with some of the constituents of the sulfated oil, and in others it may remain in its original condition. However, in either event, the purpose of this invention, namely, the production of a clear, substantially permanently stable sulfated oil, is accomplished by incorporating an organic nitrogen compound in the sulfated oil.

It has also been found that compositions prepared in accordance with this invention are suitable for treating textiles; for example, for softening textiles. This invention is therefore intended to include compositions for treating textiles and methods for preparing the same in which the problem is presented of maintaining the pH value of the sulfated oil between about 5 and about 7.5 while also preserving the clarity of the sulfated oil.

It will be evident from the above description that the compositions prepared in accordance with this invention display considerable improvement over those heretofore employed, since they may be stored for considerable periods of time without becoming cloudy, and may be used to treat leather without in any way detrimentally affecting the leather being treated.

The term "sulfated oils" used throughout the specification and in the claims is intended to refer to the sulfated oils in general use in the leather and other industries, and which ordinarily contain water, inorganic salts and, in some cases, raw oils and other substances; these oils are more commonly referred to as sulfonated oils.

Since certain changes in carrying out the above process and certain modifications in the composition which embody the invention may be made without departing from its scope, it is intended that all matter contained in the above description shall be interpreted as illustrative and not in a limiting sense.

Having described our invention, what we claim as new and desire to secure by Letters Patent, is:

1. A process of treating fibrous material which comprises contacting said material with a sulfated oil having a pH value between about 5 and about 7.5, and containing a relatively small amount of an organic nitrogen compound selected from the group consisting of amides, thiamides, and reaction products thereof with constituents of the sulfated oil.

2. A process of fatliquoring leather which comprises treating the leather with an emulsion of a sulfated oil having a pH value between about 5 and about 6, and containing between about 0.5% and about 5% of an organic nitrogen compound selected from the group consisting of amides, thiamides, and reaction products thereof with constituents of the sulfated oil.

3. A clear and substantially permanently stable sulfated oil having a pH value between about 5 and 7.5, and containing a relatively small amount of an organic nitrogen compound se-

lected from the group consisting of amides, thiamides, and reaction products thereof with constituents of the sulfated oil.

4. A clear and substantially permanently stable sulfated oil having a pH value between about 5 and about 6, and containing a relatively small amount of an organic nitrogen compound selected from the group consisting of amides, thiamides, and reaction products thereof with constituents of the sulfated oil.

5. A clear and substantially permanently stable sulfated oil having a pH value between about 5 and about 7.5, and containing between about 10% and about 50% water and a relatively small amount of an organic nitrogen compound selected from the group consisting of amides, thiamides, and reaction products thereof with constituents of the sulfated oil.

6. A clear and substantially permanently stable sulfated oil having a pH value between about 5 and about 6, and containing between about 0.5% and about 5% of an organic nitrogen compound selected from the group consisting of amides, thiamides, and reaction products thereof with constituents of the sulfated oil.

7. A clear and substantially permanently stable sulfated oil having a pH value between about 5 and about 6, and containing a relatively small amount of a compound selected from the group consisting of urea, and reaction products thereof with constituents of the sulfated oil.

8. A clear and substantially permanently stable sulfated oil having a pH value between about 5 and about 6, and containing a relatively small amount of a compound selected from the group consisting of thiourea, and reaction products thereof with constituents of the sulfated oil.

9. A clear and substantially permanently stable sulfated oil having a pH value between about 5 and about 6, and containing a relatively small amount of a compound selected from the group consisting of acetamide, and reaction products thereof with constituents of the sulfated oil.

10. A clear and substantially permanently stable sulfated oil having a pH value between about 5 and about 6, and containing between about 25% and about 30% water and between about 0.5 and about 5% of a compound selected from the group consisting of urea, and reaction products thereof with constituents of the sulfated oil.

11. A clear and substantially permanently stable sulfated oil having a pH value between about 5 and about 6, and containing between about 25% and about 30% water and between about 0.5 and about 5% of a compound selected from the group consisting of thiourea, and reaction products thereof with constituents of the sulfated oil.

12. A clear and substantially permanently stable sulfated oil having a pH value between about 5 and about 6, and containing between about 25% and about 30% water and between about 0.5 and about 5% of a compound selected from the group consisting of acetamide, and reaction products thereof with constituents of the sulfated oil.

13. A clear and substantially permanently stable sulfated oil having a pH value between about 5 and about 7.5, and containing relatively small amounts of a reducing agent and an organic nitrogen compound selected from the group consisting of amides, thiamides, and reaction products thereof with constituents of the sulfated oil.

14. A clear and substantially permanently stable sulfated oil having a pH value between about 5 and about 6, and containing relatively small amounts of sodium bisulfite and an organic nitrogen compound selected from the group consisting of amides, thiamides, and reaction products thereof with constituents of the sulfated oil.

15. In a process of preparing a sulfated oil having a pH value between about 5 and about 7.5 suitable for treating fibrous material, involving treating an oil with sulfuric acid and neutralizing the treated oil to the desired pH value, the improvement which comprises incorporating in the sulfated oil a relatively small amount of an organic nitrogen compound selected from the group consisting of amides and thiamides.

16. In a process of preparing a sulfated oil having a pH value of between about 5 and about 6 suitable for treating leather, involving treating an oil with sulfuric acid, neutralizing the treated oil to the desired pH value, and adjusting its moisture content so that the final product contains between about 25% and about 30% water, the improvement which comprises incorporating in the sulfated oil an amount of urea equivalent to between about 0.5% and about 5% of the final product.

17. In a process of preparing a sulfated oil having a pH value of between about 5 and about 6 suitable for treating leather, involving treating an oil with sulfuric acid, neutralizing the treated oil to the desired pH value, and adjusting its moisture content so that the final product contains between about 25% and about 30% wa-

ter, the improvement which comprises incorporating in the sulfated oil an amount of thio-urea equivalent to between about 0.5% and about 5% of the final product.

18. In a process of preparing a sulfated oil having a pH value of between about 5 and about 6 suitable for treating leather, involving treating an oil with sulfuric acid, neutralizing the treated oil to the desired pH value, and adjusting its moisture content so that the final product contains between about 25% and about 30% water, the improvement which comprises incorporating in the sulfated oil an amount of acetamide equivalent to between about 0.5% and about 5% of the final product.

19. A process of clearing a cloudy sulfated oil having a pH value between about 5 and about 7.5, which comprises introducing into said oil an organic nitrogen compound selected from the group consisting of amides and thiamides.

20. A process of clearing a cloudy sulfated oil having a pH value between about 5 and about 6, and containing between about 25% and about 30% water, which comprises introducing into said oil an amount of a reducing agent equivalent to between about 0.25% and about 1% of the final product, and an amount of an organic nitrogen compound selected from the group consisting of amides and thiamides equivalent to between about 0.5% and about 5% of the final product.

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