This invention relates to the conversion of chemical cellulose pulp into alkali cellulose in a steeping operation with an aqueous solution of sodium hydroxide (steeping liquor) and has for its object the provision of an improved process for the formation of alkali cellulose in slurry steeping. "Chemical cellulose pulp," as the term is used herein, refers to wood pulp, cotton linter pulp, or mixtures thereof, which are used in chemical conversions or in chemical processing. The slurry-steeping of chemical cellulose pulp is usually carried out by feeding small rectangular sheets or continuous sheets of the cellulose pulp into a suitable vessel containing steeping liquor, containing, say, from 16% to around 20% of sodium hydroxide in which a mechanically operated device, such as paddles or blades, beat the cellulose into a slurry. The operations are frequently carried out at cellulose consistencies of around 2% to 6% and at temperatures of about 20° to 50°C.

While such steeping operations are commonly carried out to form alkali cellulose for use in the viscose process they may also be carried out to form alkali cellulose for use in other processes, such as in the production of hydroxyethyl cellulose. In the slurry-steeping of such chemical cellulose pulp considerable difficulties have been encountered mainly due to foaming of the steeping liquor and floating and clumping of the fibers which have a deleterious effect in the steeping and pressing operations as well as in subsequent processing. It appears that small bubbles of gas (presumably air) form on the fibers of pulp causing the undesirable foaming, floating, flocculation, and clumping.

Chemical cellulose pulp formed from wood usually contains up to 0.35% (based on the dry weight of the pulp) of residual natural and other extractable resins. Highly refined chemical cellulose pulp usually contains up to 0.15% of such resins. This ether extractable matter, loosely called "resins," is mainly in the nature of waxes, fats and true resins, the latter often being present in small amount, a part being saponifiable and a part being unsaponifiable. In the production of chemical cellulose pulp which is a highly refined wood pulp it is not practical to remove all the resins from the pulp.

Some of these resins have certain specific surface active properties or form with the sodium hydroxide in the steeping operation soaps that have certain surface active properties and these may cause or contribute to the cause of foaming, floating and clumping. The fibers of the pulp may also have certain physical properties such as inherent kinkiness which cause an affinity for gas bubbles, presumably small bubbles of air, to be formed on and entrapped within the mass of fibers. These possible theories are offered as a plausible explanation of the objectionable conditions encountered in the slurry-steeping of such pulp. If any satisfactory method can be found for overcoming these difficulties the demand for alkali cellulose will be increased.

We have made the surprising discovery that certain water-soluble substantially non-ionic surfactants have the property of substantially or completely overcoming these objectionable conditions when incorporated in the slurry-steeping constituents, either by addition to the pulp or in the steeping liquor. Such compounds for use in our process are water soluble substantially non-ionic compounds which consist of the products of a hydrophobic compound of low water solubility containing at least 2 active hydrogens substituted by at least 2 terminal polyethylene oxide groups each containing from 1.6 to 700 ethylene oxide units. The compounds used in the process of the invention may be represented by the formula:

\[ R - \left[ \text{OCH}_2\text{CH}_2\text{OH} \right]_n \]

wherein \( R \) is a nucleus of an organic hydrophobic compound of low water solubility having from 2 to 4 active hydrogens and in which \( x \) varies from 1.6 to 700 and \( n \) equals 2 to 4.

When \( R \) is \( -\left(\text{OCH}_3\right)_3\text{CH} \), \( y \) varies from 19 to 31, and has two terminal polyethylene oxide groups the compound is of the class known as poly(vinyl) block copolymers. The term "block copolymers," as used herein, will be understood to be the block copolymers of polyethylene oxide and polypropylene oxide which may be represented by the formula:

\[ \text{HOCH}_2\text{CH}_2\text{O}(\text{CH}_2\text{CH}_2\text{O})_m\text{CH}_3 \]

in which \( x \) and \( y \) have average values for particular copolymers of 10 and 19; 3.3 and 28; 6 and 28; 15.5 and 28; and 106 and 28, respectively. In one especially effective block copolymer, the average values of \( x \) and \( y \) are 15.5 and 28 respectively, \( y \) varying from 11 to 17 depending upon the total molecular weight, and the value of \( y \) varying from 26 to 31 depending upon the total molecular weight and the value of \( x \). Block copolymers of the aforementioned types are manufactured and sold under the names of Pluronic L-44, Pluronic L-61, Pluronic L-62, Pluronic L-64, and Pluronic F-68, Pluronic L-64, which is believed to have average values for \( x \) and \( y \) of 15.5 and 28 respectively, is outstandingly effective for the purpose of the invention.

\( x \) is also represented by the nucleus:

\[ (\text{OC})_n\text{H}_2 \]

in which \( y \) represents average values of from 3 to 17.2. When said nucleus \( R \) is combined with four polyethylene oxide radicals the compound is represented by the formula:

\[ \text{HOCH}_2\text{CH}_2\text{O}(\text{CH})_n\text{CH}_3 \]

in which \( x \) and \( y \) have average values of from 2.8 to 437 and 3 to 17.2 respectively, the compound is effective for use in the process of the invention. Such compounds are sold under the trademark Tetrone. Tetrone 304, 504, 701, 702, 704, 707, 904 and 908 having average values of \( x \) and \( y \) of 3.5 and 3.0, 8.1 and 7.5, 2.8 and 11.6, 5.2 and 11.6, 12.8 and 11.6, 47.0 and 11.6, 18.3 and 11.6.
and 17.2, and 437 and 17.2 respectively, are especially suitable for use in the invention.

The foregoing compounds may be identified further with reference to the following table:

<table>
<thead>
<tr>
<th>Compound</th>
<th>Molecular Weights</th>
<th>Percent Ethylene Oxide</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Range</td>
<td>Average</td>
</tr>
<tr>
<td>Pluronic L-44</td>
<td>1,657-2,400</td>
<td>2,000</td>
</tr>
<tr>
<td>Pluronic L-55</td>
<td>1,667-2,580</td>
<td>2,020</td>
</tr>
<tr>
<td>Pluronic L-64</td>
<td>2,500-3,000</td>
<td>3,000</td>
</tr>
<tr>
<td>Pluronic F-68</td>
<td>7,600-10,000</td>
<td>8,000</td>
</tr>
<tr>
<td>Pluronic P-75</td>
<td>3,500-5,250</td>
<td>4,353</td>
</tr>
<tr>
<td>Tetroxin 561</td>
<td>2,350-2,600</td>
<td>2,500</td>
</tr>
<tr>
<td>Tetroxin 644</td>
<td>2,500-4,000</td>
<td>3,182</td>
</tr>
<tr>
<td>Tetroxin 703</td>
<td>2,777-3,730</td>
<td>3,235</td>
</tr>
<tr>
<td>Tetroxin 707</td>
<td>3,125-4,266</td>
<td>3,667</td>
</tr>
<tr>
<td>Tetroxin 708</td>
<td>4,165-5,000</td>
<td>4,500</td>
</tr>
<tr>
<td>Tetroxin 707</td>
<td>8,334-15,000</td>
<td>11,000</td>
</tr>
<tr>
<td>Tetroxin 688</td>
<td>6,600-9,000</td>
<td>7,254</td>
</tr>
<tr>
<td></td>
<td>36,000-150,000</td>
<td>81,000</td>
</tr>
<tr>
<td></td>
<td>90-100</td>
<td></td>
</tr>
</tbody>
</table>

In carrying out a process of the invention the surfactant may be incorporated in the slurry in any suitable way, for example, it may be most conveniently added to the chemical cellulose pulp during its preparation, or to the steaming liquor or added to the slurry. Only a relatively small amount of the surfactant is necessary, varying from about 0.02% to 0.25% by weight based on the dry weight of the pulp. Amounts of the surfactants in excess of 0.25% are not believed to be detrimental, but only unnecessary. As used herein, the word "dry" means substantially dry and refers to pulp either bone dry or containing that small moisture content (e.g. 6%) which the pulp, being a hygroscopic substance, has taken up from the air. Roughly, this will refer to pulp containing from zero to 10% of moisture.

The accompanying drawing was reproduced from a photograph of samples of chemical cellulose taken of pulp undergoing slurry-steeping. The photograph is of glass vessels taken against a black background; the white part is the alkali cellulose while the dark part is the steaming liquor from which the alkali cellulose has separated.

Each of the samples of pulp shown in the drawing was slurry-steeped by mechanical treatment of identical pulp sheets of 96% alpha cellulose containing 0.12% ether extractable matter. The pulp was slurried at 22.2% consistency in a steaming liquor containing 18.6% sodium hydroxide and 1.5% hemicellulose at 45°C. In each of the slurry samples shown in the drawing, 0.1% of a surfactant based on the dry weight of the pulp was incorporated in the slurry as follows and the samples were left at rest for four hours:

- (A) Tetrionic 304
- (B) Tetrionic 504
- (C) Tetrionic 701
- (D) Tetrionic 702
- (E) Tetrionic 704
- (F) Tetroxic 707
- (G) Tetroxic 904
- (H) Tetroxic 908
- (I) Pluronic L-64
- (J) Control

The aforementioned Tetrionics and the Pluronics, are very effective surfactants in the process of the invention. The very effective improvement in slurring is shown by the drawings in which it will be noted that the upper surfaces of the samples A to I are about at the same height and flat on top in comparison with the elevated or floated pulp of the control which has an irregular top. Moreover, samples A to I show no appreciable separation of pulp from the water while the control shows very appreciable separation of the pulp from the water.

When the cellulose is slurred to form alkali cellulose for conversion to viscose the surfactants are especially effective because they do not cause adverse effects in the subsequent viscose process operations. Some of the other steps in the viscose process are actually improved by the use of these surfactants.

Jordmann or other mechanical treatment of pulp fibers is helpful in straightening out kinky fibers and making them less susceptible to entrapment or retention of air when slurried in solutions of strong caustic soda.

Such treatment makes it possible to use smaller amounts of added surfactant and still obtain smooth slurries that do not foam or clump. In another aspect, use of such surfactants may be made in reducing floating and clumping of pulp fibers in pulp refining operations in which the cellulose fibers are being extracted, purified or washed with solutions containing sodium hydroxide or other alkaline agents.

We claim:

1. The process of slurry-steeping chemical cellulose pulp containing up to 0.33% based on the weight of the pulp of natural ether extractable matter in aqueous sodium hydroxide solution to form alkali cellulose, comprising carrying out the slurry-steeping with from about 0.2% to about 0.25% based on the weight of the dry pulp of an added surfactant in the steeping solution and substantially eliminating foaming, floating, flocculation and clumping of the pulp during the slurry-steeping, said surfactant being a water soluble substantially non-ionic compound which consists of the product of a polypropylene oxide containing hydrophobic compound of low water solubility containing at least 2 active hydrogens substituted by at least 2 terminal polyethylene oxide groups each containing from 1.6 to 700 ethylene oxide units.

2. In the process of claim 1, using as a surfactant a block copolymer having the formula:

\[ RO(CH_2CH(OH)_2)_n(CH_2CH_2O)_mH \]

in which the average values of x and y vary from 3.3 to 106 and from 19 to 31 respectively.

3. In the process of claim 1, using as a surfactant a block copolymer having the formula:

\[ RO(CH_2CH(OH)_2)_n(CH_2CH_2O)_mH \]

in which the average values of x and y are about 15.5 and 28 respectively.

4. In the process of claim 1, using as a surfactant a compound having the formula:

\[ HO(CH_2CH(OH)_2)_n(CH_2CH_2O)_mH \]

in which x and y have average values of from 2.8 to 437 and 3 to 17.2 respectively.

5. In the process of claim 1, using as a surfactant a compound represented by the formula:

\[ HO(CH_2CH(OH)_2)_n(CH_2CH_2O)_mH \]

in which x and y have average values of about 3.5 and 3.0 respectively.

6. In the process of claim 1, using as a surfactant a compound represented by the formula:

\[ HO(CH_2CH(OH)_2)_n(CH_2CH_2O)_mH \]

in which x and y have average values of about 12.8 and 11.6 respectively.

7. In the process of claim 1, forming the slurry of a
wood pulp which had been subjected to a mechanical
operation to straighten the fibers and thereby reduce the
amount of surfactant required.

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