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WOOD PULP

Kenneth Russell Gray, Shelton, Wash., assignor to Rayonier Incorporated, Shelton, Wash., a corporation of Delaware

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This invention relates to chemically prepared wood pulp such as the so-called "dissolving pulp" and hereinafter, for convenience, will be called "refined wood pulp." The invention has for its object the provision of an improved refined wood pulp and a method of producing the same.

Refined wood pulp consists mainly of cellulose with relatively low non-cellulosic impurities, such as hemi-cellulose, fats, resins, waxes, high molecular weight alcohols, etc. A substantial portion of the non-cellulosic impurities is extractable with ether, and is hereinafter called the "ether extract" or the "ether extractable matter."

I have found that the ether extract contains two distinctly different types of materials which possess or can exert under appropriate conditions interfacially active properties and which have important effects on the processing qualities of the pulp. These two classes are:

1. The saponifiable portion of the ether extract (otherwise termed ether extractable saponifiable matter) which is of the nature of fatty and resin acids either in the free state or combined in such forms as glycerides or esters of higher alcohols. These materials in their saponified state are water soluble soaps, i. e., are anion active agents. (Where numerical values for saponifiable matter are herein given, such values refer to the total amount of fatty and resin acids, both in free and combined form).

2. Another portion consisting of unsaponifiable materials which are substantially non-volatile, insoluble in water and dilute caustic soda, soluble in oil, and interfacially active at an oil-water interface. These unsaponifiable oil soluble, interfacially active materials are in general high molecular weight alcohols such as phytosterol. (Phytosterol is the generic name for sterols derived from vegetable sources. Thus a sample of "phytosterol" may be a chemical individual or a mixture of closely related sterols).

Part of the phytosterol is in the form of saponifiable esters with higher fatty acids. On saponification these esters form soaps and liberate free phytosterol. The phytosterol itself is, however, unsaponifiable whether in free or combined form. Furthermore, in this connection in referring to the unsaponifiable portion of the ether extract (otherwise termed "ether extractable unsaponifiable matter") there is meant all the water insoluble residue which in a saponification treatment does not combine with caustic alkalies to form water soluble soaps, irrespective as to whether such material is in free or combined form in the ether extract.

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As commonly used and as referred to specifically herein, the term "interfacially active" applied to a material refers to the material being capable of lowering the interfacial tension at an interface by virtue of a positive adsorption therein. The term "surface active" material refers to a water soluble material which will specifically reduce the interfacial tension at an air-water interface, such a liquid-air interfacial tension being commonly termed the "surface tension". By the term "anion active" is meant a water soluble, ionizable surface active (and thus interfacially active) material, the surface activity of which is due to the anion.

The unsaponifiable, oil soluble, interfacially active agents of the invention constitute a large fraction of the unsaponifiable portion or residue of the ether extract. The unsaponifiable ether extractable matter also contains other fractions such as hydrocarbons and chlorinated hydrocarbons which are not interfacially active. Nevertheless, the amount of total unsaponifiable ether extractable matter serves generally as a relative indication of the amount of unsaponifiable, oil soluble, interfacially active material and is a more easily determined value.

I have found that as regards different woods commonly used for producing pulp such as pine, spruce, and hemlock that the ether extract left in the refined pulp varies appreciably in the content of unsaponifiable matter and thus also as regards the proportions of oil soluble unsaponifiable interfacially active material and of saponifiable matter which will be interfacially active in the water soluble, saponified state. A comparison of the compositions of ether extracts from refined pulps prepared from different woods and having the same total amounts of ether extract follows:

Comparison of composition of ether extract of refined chemical pulp prepared from different woods

Wood Used in Preparing Pulp	Total Ether Extract	Per Cent of Material Present Based on Bone Dry Pulp	
		Saponifiable Matter	Unsaponifiable Matter
Hemlock	.10	.0566	.034
Spruce	.10	.0484	.0405
Southern Pine	.10	.072	.0144

Such figures as given above are also somewhat dependent on the process by which the pulp is made.

The two classes of extractable materials which are interfacially active or which can become interfacially active have different physical and chemical properties and exercise different effects in the subsequent utilization of the pulp, as in its conversion into various cellulosic products in the chemical and rayon industries. Some of these effects are desirable while others are not, but uniformity of effect is very necessary.

I have discovered that the behavior of the refined wood pulp in its conversion into various cellulosic products cannot be predicted on a basis of the amount of residual ether extract considered as a whole. I have found, for example, that hemlock and pine pulps each having the same quantity of ether extract, say 0.15%, do not have the same properties because the hemlock pulp will contain a higher proportion of ether extractable, unsaponifiable matter. The hemlock pulp thus contains a higher proportion of oil soluble unsaponifiable interfacially active materials and a lower proportion of saponifiable matter, interfacially active in the water soluble, saponified state.

It is accordingly one of the objects of my invention to provide a refined wood pulp containing uniform quantities of either saponifiable matter, interfacially active when saponified, or unsaponifiable interfacially active ether extractable matter, or both, regardless of the kind of wood from which the pulp was produced.

In accordance with the method of my invention, I attain uniformity of pulp composition with respect to ether extract by ascertaining the kinds and proportions thereof in the pulp and then adding the required amount of oil soluble unsaponifiable interfacially active materials or saponifiable fatty or resin acid soap forming materials (either as such or as their effective equivalent in the form of saponified soaps), the additions being in the form of selected compounds to produce a pulp with a uniform amount of either one or both together as required.

The refined wood pulps, such as the so-called "dissolving pulps", usually contain less than 0.07% of unsaponifiable ether extractable matter. The invention is especially and advantageously applicable to those pulps containing a relatively low percentage of unsaponifiable ether extractable matter, say less than 0.02%, wherein substantial additions are necessary to attain a predetermined norm of uniformity for all species of refined wood pulp.

As heretofore pointed out, the amount of unsaponifiable ether extractable matter has been chosen as a standard rather than the exact amount of unsaponifiable ether extractable interfacially active matter in that the former serves as a relative indication of the amount of the latter and may be much more readily determined.

While it will be readily obvious to anyone skilled in the art of how to determine ether extractable unsaponifiable matter, nevertheless for clarity I give the standard procedure which follows: 350 gm. of pulp of known moisture content, usually about 4–8% moisture, is extracted 8 hours with ether in a Soxhlet extractor. The ether is evaporated off the extract solution and the residue may be weighed if desired to determine the total amount of ether extractable matter. The residue from the ether evaporation is then heated with 100 cc. normal alcoholic KOH for 1 hour. The solution is then transferred to a separatory funnel, an equal quantity of water added and the unsaponifiable matter extracted with ether. The ether solution is washed with water, dried by addition of anhydrous Na_2SO_4, after which the solution is filtered, the sodium sulphate being discarded after being washed with ether. The ether is evaporated off and the residue heated one hour at 105° C., then cooled and weighed. The value for ether extractable unsaponifiable matter so obtained is then calculated as the per cent on bone dry pulp.

The unsaponifiable water insoluble, oil soluble compounds, interfacially active at an oil-water interface, which I add in practicing my invention, like conventional water-soluble surface active materials, are hydrophilic-lipophilic combinations. These compounds have a strong lipophilic group or groups, substantially of a hydrocarbon character, together with a group or groups having a relatively weak hydrophilic effect. Thus, the compounds used in my invention are at least slightly oil soluble but substantially water insoluble. While the hydrophilic effect is not enough for the compounds to be appreciably soluble in water, there is nevertheless a substantial attraction for water. Hence, the compounds will tend to concentrate wherever there is an oil-water interface with the lipophilic portion dissolved in the oil and the hydrophilic portion dissolved in the water.

The unsaponifiable interfacially active compounds used in my invention do not greatly lower the surface tensions of any oils they dissolve in. Being substantially insoluble in water or aqueous solutions, they do not appreciably lower the surface tension of aqueous phases. Nevertheless, when present at an oil-water interface, they produce very low interfacial tensions.

As heretofore stated, the unsaponifiable interfacially active compounds used in the invention are substantially non-volatile, oil soluble, substantially water insoluble materials, interfacially active at an oil-water interface. In general these compounds are high molecular weight alcohols, i. e., alcohols containing at least 10 carbon atoms. The alcohols may be aliphatic (straight or branched chain, saturated or unsaturated) or cyclic. They may be primary, secondary, or tertiary. The alcohols moreover may be either mono or dihydroxylic. I prefer to use high molecular weight cyclic alcohols and of these have found sterols to be particularly effective, especially phytosterols.

Examples of unsaponifiable, oil soluble, interfacially active compounds I have found to be effective follow:

1. Aliphatic Monohydroxylic Alcohols:
   n-oleyl, n-dodecyl, n-cetyl, n-stearyl alcohols.
   5-ethylnonanol-2, 7-ethyl-2-methylundecanol-4 and 3,
   9-diethyltridecanol-6.
2. Aliphatic Glycols:
   Octadecanediol-1, 12.
3. Cycloaliphatic Alcohols Derived from Rosin:
   Dihydroabietyl, dehydroabietyl, tetrahydroabietyl, and abietyl alcohols. Mixtures of dihydroabietyl, dehydroabietyl, tetrahydroabietyl, and abietyl alcohols of which one is sold under the trade name of "Hydroabietyl" alcohol.
4. Sterols and Sterol Containing Materials:
   Phytosterols, stigmasterol, sitosterol, ergosterol, α-tocopherol, chloesterol, lanolin, hemlock pulp ether extract unsaponifiables, spruce pulp ether extract unsaponifiables.

The addition to and incorporation of the un-

saponifiable oil soluble, interfacially active compounds in the wood pulp may be effected in any suitable way, providing, of course, the distribution is adequately uniform. In those operations in which the pulp is formed into sheets, additions may advantageously be subsequent to sheet formation, but prior to complete drying, by spraying the pulp sheet with an aqueous emulsion or dispersion of the compound. The sheet may be passed over a rotating roll partly immersed in such an emulsion or dispersion. If desired, the unsaponifiable oil soluble, interfacially active compounds used in the invention may be incorporated in the pulp prior to sheet formation as, for example, by making additions to a suspension of pulp in water in a stock chest.

The application of the compounds to the pulp may be accomplished by use of a solution of the compound in a satisfactory organic solvent but more practically by use of an aqueous emulsion as described above. The preparation of such an aqueous emulsion may be effected by an appropriate procedure common to the art, generally in the presence of a small amount of a water soluble emulsifying agent.

Where the ether extract of the pulp is very low in addition to adding an unsaponifiable, oil soluble interfacially active material, there may also be added according to Patent Re. 22,489, fatty or resin acid soap forming materials or their effective equivalents in the form of the anion active, water soluble saponified soaps. Also other anion active agents may be added according to Re. 22,489. When such materials are added in a water soluble, anion active condition, it will frequently be convenient to emulsify the unsaponifiable, oil soluble interfacially active materials in a solution of the anion active compound. Of the anion active or potentially anion active compounds, the most suitable are the saponifiable soap forming materials (fatty or resin acid materials) or their effective equivalents in the form of the saponified soaps.

While the unsaponifiable oil soluble, interfacially active compounds used in this invention are especially suitable for use in combination with anion active or potentially anion active compounds, they are also suitable for use in combination with cation-active compounds, non-ionic surface active compounds and mixtures of these.

Since the amount of unsaponifiable ether extractable matter varies widely in pulps from different woods, the invention makes it possible to impart to pulp from one wood characteristics of pulp from a different wood. In this way, I can produce a uniform refined wood pulp, in other words, an industrially uniform composite irrespective of the many different kinds of woods from which it originates.

I claim:

1. In a method of producing an improved wood pulp from a refined wood pulp containing not over 0.15% of natural ether extractable matter consisting of saponifiable matter and not over 0.07% of oil soluble unsaponifiable matter, ascertaining the quantities of saponifiable and unsaponifiable matter in the pulp and adjusting the percentages thereof to a predetermined norm, not over 0.15%, by the addition to the pulp of at least one of the said forms of ether extractable matter, the saponifiable addition being a compound of the group consisting of fatty acid and resin acid which when saponified is a water soluble soap, and the unsaponifiable addition being an oil soluble alcohol selected from the group consisting of an aliphatic alcohol containing at least 10 carbon atoms and a cycloaliphatic alcohol containing at least 10 carbon atoms whereby pulp having uniform characteristics is produced from different types of woods.

2. In the method of claim 1, incorporating in the wood pulp a monohydroxylic alcohol to produce a refined wood pulp having more unsaponifiable matter than the original pulp.

3. In the method of claim 1, incorporating in the wood pulp a sterol to produce a refined wood pulp having more unsaponifiable matter than the original pulp.

4. In the method of claim 1, incorporating in the wood pulp cholesterol to produce a refined wood pulp having more unsaponifiable matter than the original pulp.

5. In the method of claim 1, incorporating in the wood pulp phytosterol to produce a refined wood pulp having more unsaponifiable matter than the original pulp.

KENNETH RUSSELL GRAY.

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