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JOSEPH OTIS PEIRCE AND WARREN T. REDDISH, OF CINCINNATI, OHIO, ASSIGNORS TO THE TWITCHELL PROCESS COMPANY, OF CINCINNATI, OHIO, A CORPORATION OF OHIO.

PROCESS OF ISOLATING CELLULOSE.

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Our invention relates to the art of isolating cellulose and is particularly directed to the isolation of cellulose in paper making. The invention is illustrated by the following disclosure of a process of making paper using used paper as a raw material.

Problems.

Making paper out of used paper involves the following accomplishments:

1. Re-pulping the paper, that is, disintegrating the paper into a fibrous, pulpy mass.

2. Disassociating the foreign matter from the fibre. The foreign matter may be discoloring matter, dirt, starch, glue, or it may be ink, such as printer's ink, the problem being particularly acute when the foreign matter is ink. This disassociating involves two accomplishments, (*a*) debonding foreign matter from the fibre or cellulose, (*b*) separating or segregating the foreign matter and the cellulose so that the cellulose is isolated.

Objects.

One object of the invention is to provide a process of isolating cellulose.

Another object of the invention is to provide a process of making new paper out of used paper.

Another object of our invention is to provide a process of disassociating foreign matter from paper fibre.

Another object of our invention is to provide a process of making paper in which process the disassociation of the foreign matter from the paper fibre and the bleaching of the pulp are advantageously correlated.

Another object of our invention is to provide a process of deinking print.

Machinery.

In the following description of our invention no particular machinery is described or called for since the process is adapted to be carried on with the conventional machinery already utilized in paper making. This machinery comes under the general classifications of pulping equipment or beaters, washing apparatus and bleaching apparatus. But it must be understood and remembered, throughout the description of this invention, that time factors, temperatures and concentration of reagents are variable, depending on the nature and character of the used paper under treatment, the character of paper desired, and the efficiency of the apparatus used.

General process.

The invention comprises reducing the used paper to a pulp, subjecting the fibre to the action of special chemical reagents, washing the pulp, bleaching (if necessary or desirable), and finally making paper out of the pulp according to the conventional processes.

Description of reagents.

The chemical reagents which we use in this process are variously procurable, and of complex and uncertain structure chemically. Therefore a rather detailed description of these chemical reagents is provided. Our reagents comprise derivatives of the products formed by treating certain oils with sulphuric acid. The oils embraced within the scope of this invention are those hydrocarbon oils characterized by the presence of certain components susceptible to modification by the action of sulphuric acid and certain components not susceptible. Oils such as petroleum, shale oil and coal tar oil, gilsonitic distillates and many other fractions or distillates of hydrocarbon deposits usually exemplify this characteristic.

Since petroleum is commercially available at present in large quantities, the following disclosure of our reagents is made in relation thereto.

When petroleum or fractions thereof, such as lubricating stock, are treated with sulphuric acid, certain components of the oil are modified. The theory chemically accepted at present explains this modification as sulfonation, i. e. the sulphuric acid reacts with components of the mineral oil (the unsaturated components, supposedly) to form mineral oil sulphonic acid. The yield of sulphonic acid is higher when the oil is treated with fuming sulphuric acid, so this disclosure is also made in relation to this fuming sulphuric acid treatment.

It is further theory that these mineral oil sulphonic acids can be reacted with metallic substance or compounds such as sodium hy-

droxide or sodium carbonate, for instance, to produce salts of mineral oil sulphonic acid. Research to date seems to indicate that these assumptions are correct, but in the ensuing description of the invention and the claims, it is to be understood that the procedure and products thereof as described is the real substance of the invention and that the chemical explanation is merely offered for the benefit of the skilled in the art.

This above described sulphonating treatment is practiced on a large commercial scale in the refining of medicinal white oil. In this refining operation the sulphonated components are the by-products and are commercially available as such at present. These sulphonated components are usually available in the form of alkaline salts since the sulphonated components in the acid state have a corrosive action on iron or steel pipes or containers. The advantage of treating, shipping or handling these sulphonated components in a neutral state is a continuing one.

In commercial practice a mineral oil fraction such as a lubricating stock is treated with fuming sulphuric acid. Two layers are formed. The upper layer is known as the A layer or oil layer and the lower layer as the B layer or sludge layer.

The process of refining the white oil and extracting the sulphonates of the oil layer is described and claimed in U. S. Patent No. 1,087,888 to Grigori Petroff, issued February 17, 1914; U. S. Patent No. 1,233,700 to Grigori Petroff, issued July 17, 1917; U. S. Patent No. 1,196,274 to Grigori Petroff, issued August 26, 1916, and a specific process in U. S. Patent No. 1,286,179 to Robert E. Humphries, issued November 26, 1918.

Some of the pertinent sulphonic sludge layer acids and salts, and the processes of extracting or making them are described in the patents to Robert E. Divine, namely:

1,301,662 Apr. 22, 1919; 1,301,663 Apr. 22, 1919; 1,303,779 May 13, 1919; 1,319,027 Oct. 14, 1919; 1,330,624 Feb. 2, 1920; 1,438,101 Dec. 5, 1922.

All of these sulphonic acids and derivatives of sulphonic acids are either gummy viscuous liquids or solids, and according to chemical theory are characterized by very high molecular weights as compared with benzine sulphonic acid.

It is recommended that these sulphonic bodies be used in a state as pure as possible especially with regard to oil because oil decreases the effectiveness of these reagents in regard to their action in reducing the adhesion of printer's ink to paper fibre.

Selection of paper.

Paper must first of all be freed from grease, leather or cloth book covers, books with burned edges, rust stained paper, rubbish, string, leather, rags, wire, wood, etc. This is done by assorting the waste or used paper, and it is also recommended that the used paper be graded according to quality to control the uniformity of the new paper to be made therefrom.

Repulping.

The used paper is then beaten with water, in a beating machine. The object of the beating operation is to provide continuous agitation and break down the paper into pulp. Machines other than beating machines (technically) can be used if they accomplish the desired objective. The instant process is adapted to be used with any repulping or defibring devices.

Disassociation of foreign matter.

It is preferable to combine this repulping or new pulping of the paper with the step of debonding the foreign matter from the fibre. The sulphonic reagent is therefore added to the water used in the defibring operation prior to the introduction of the used paper which is then beaten and boiled in the aqueous sulphonic solution until the foreign matter is debonded. The sulphonic derivatives of the class described have a debonding effect and solvent action on foreign matter in general including sizing, glue starch, dirt, and printer's ink, particularly at elevated temperatures such as 200°–212° F. and do not harmfully attack the cellulose or shorten the fibre. Aqueous sulphonic solutions from a small fraction of 1% up (sulphonic body to water) have solvent action on printer's ink, but solutions between ½% and 4% are recommended. The ½% solution will remove the ink and other foreign matter from the paper boiled in said solution, in about forty minutes, one pound of sulphonic reagent being adequate to treat twenty-five pounds of paper.

After the foreign matter has been disassociated from the pulp, the foreign matter and the pulp are separated, for instance by drawing off the sulphonic solution and washing the paper by agitation, with clean water, at a temperature such as 200°–212° F. The pulp is then ready to be reclaimed and made into new paper. If necessary the pulp can be bleached before being made into paper but this depends on the stock treated and the use to which the new paper is to be put.

Selection of reagent.

The choice of particular sulphonic reagent adapted for the needs of the user must be determined by the skilled in the art. This is because of variations in paper stock, water (quality and quantity available), machinery, general equipment, and the intended use for the new paper. The sulphonic sludge products are recommended in preference to the oil layer sulphonic products because of their

greater water solubility. The California sludge products are particularly recommended since the calcium and magnesium salts thereof are more readily water soluble and could therefore be more easily used in hard water.

The use of sludge layer sodium sulphonate is particularly recommended because of the tendency of this body to hold in suspension or solution otherwise insoluble organic and inorganic salts. The use of this body therefore prevents some of the deposits otherwise incidental to the paper making industry in addition to its de-inking effect on the pulp.

These sulphonic reagents can be used in alkaline, acid or neutral state. Too much alkalinity tends to disintegrate some fibres and is therefore to be avoided. The neutral sodium sulphonate provides good results with small deposit when used with hard water. The sulphonic acid reagent in addition to debonding the foreign matter before mentioned, provides a solution which also dissolves discoloring matter and has therefore a bleaching effect on the cellulose when used either alone or in conjunction with chlorine gas bleaching. The sulphonic reagents can be used partially neutralized or completely neutralized. Sludge layer sodium sulphonate plus a small amount of free sulphonic acid provides an adequate reagent for the above described purposes.

Thus we provide a process of isolating cellulose, said process isolating the same to a bleaching point when the acid sulphonic reagent is used.

Having described our invention, we desire to be limited only by the ensuing claims:

1. The process of making new paper out of used paper, comprising, repulping the used paper, disassociating the foreign matter therefrom by treatment with a mineral oil sulphonic reagent, washing the pulp, and making new paper from the same.

2. The process of making new paper out of used paper, comprising, repulping the used paper, disassociating the foreign matter therefrom by treatment with a sulphonic reagent, bleaching in the presence of a sulphonic acid derivative, washing the pulp, and making the same into new paper.

3. In the process of making new paper out of old paper, the step of reducing the adhesion of foreign matter to the fibre by heating the same with a mineral oil sulphonic reagent.

4. The process of de-inking print, comprising, the step of treating the paper with a mineral oil sulphonic reagent in aqueous solution, and the step of washing the paper.

5. In the art of de-inking print, the step of simultaneously reducing the paper to a pulp, and subjecting the paper to the action of a mineral oil sulphonic reagent.

6. The process of making new paper out of used paper with print thereon, comprising, repulping the used paper, disassociating the foreign matter therefrom by treatment with a sludge layer mineral oil sulphonic reagent, washing the pulp, and making new paper from the same.

7. The process of making new paper out of used paper with print thereon, comprising, repulping the used paper, disassociating the foreign matter therefrom by treatment with a sulphonic reagent, bleaching in the presence of a sulphonic sludge acid, washing the pulp, and making the same into new paper.

8. In the process of making new paper out of old paper, the step of reducing the adhesion of foreign matter to the fibre by heating the same with a mineral oil sulphonic sludge reagent.

9. The process of de-inking print, comprising, the step of treating the paper with a sludge layer mineral oil sulphonic reagent in aqueous solution, and the step of washing the pulp.

10. In the art of a de-inking print, the step of simultaneously reducing the paper to a pulp, and subjecting the paper to the action of a sludge layer mineral oil sulphonic reagent.

11. The process of making new paper out of used paper having print thereon, comprising, repulping the used paper, disassociating the foreign matter therefrom by boiling the same with a water soluble mineral oil sulphonic reagent for approximately forty minutes, washing the pulp, and making new paper from the same.

12. The process of making new paper out of used paper having print thereon, comprising, repulping the used paper, disassociating the foreign matter therefrom by boiling with a water soluble sulphonic reagent, bleaching in the presence of a sulphonic acid, washing the pulp, and making the same into new paper.

13. In the process of making new paper out of old paper, the step of reducing the adhesion of foreign matter to the fibre by boiling, and agitating the same with a mineral oil sulphonic reagent.

14. The process of de-inking print, comprising, the step of treating the paper with an oil free mineral oil sulphonic reagent in aqueous solution, and the step of washing the paper.

15. In the art of de-inking print, the step of simultaneously reducing the paper to a pulp, and subjecting the paper to the action of an oil free mineral oil sulphonic reagent.

16. The process of making new paper out of used paper, comprising, repulping the used paper at elevated temperatures, debonding the foreign matter by treatment with a mineral oil sulphonic derivative in water so-

lution, washing the pulp with hot water, and making new paper out of same.

17. The process of making new paper out of used paper, comprising, repulping the used paper at 95°–100° C. in a dilute solution of a mineral oil sulphonic derivative, debonding the ink, size, glue starch or other foreign material from the fibre, washing the cleaned pulp, and reclaiming same for use in the manufacture of new paper.

In witness whereof, we hereunto subscribe our names.

JOSEPH OTIS PEIRCE.
WARREN T. REDDISH.