

PATENT SPECIFICATION

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PROVISIONAL SPECIFICATION.



Improvements in and relating to the Manufacture of Cellulose Ethers.

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, a Company registered under the laws of Great Britain, of Broadway Buildings, Westminster, London, S.W.1, and DAVID TRAILL, a British subject, of "Cronulla", Ardrossan Road, Saltcoats, Ayrshire, Scotland, do hereby declare the nature of this invention to be as follows:—

This invention relates to cellulose derivatives, and more especially to aralkyl celluloses generally, for example, benzyl cellulose, with reference to which latter body the invention will be more particularly described.

In investigations on benzyl cellulose, particularly in connection with lacquer production, we have found that the normal type of benzyl cellulose produced gives somewhat opalescent solutions, and lacquers made from the solutions dry with a matt surface. While such a surface might be utilised, an effort has been made to obtain water clear solutions which would dry giving a highly glossy surface. The cause of the opalescence has therefore been examined, and it appears to be due to the presence of ethers which are less degraded than the bulk of the benzyl cellulose.

The object of the present invention is to provide a method for lowering the viscosity of benzyl cellulose solutions and for the modification of benzyl cellulose, so that it will give clear solutions from which may be obtained lacquers that will dry with a highly polished surface, whilst at the same time retaining the advantage of the use of cheap solvents or mixtures of solvents, such as, for example, benzene spirits or toluene spirits.

The invention consists in the treatment of benzyl cellulose or other aralkyl cellulose, which is wholly or partially insoluble in the usual organic solvents, with benzyl chloride or other aralkyl halide, and with water and steam at normal or increased pressure.

The invention also consists in a process for the working up of a reaction mass such as may be obtained by the action of benzyl chloride on alkali cellulose, as described in our co-pending Application

(Serial No. 327,714) No. 28,356/28, which comprises the purification of the reaction mass until it contains 3 to 25% of benzyl chloride or other aralkyl halide and the conversion of the latter to benzyl alcohol.

The invention also consists in the application of products such as may be formed by the processes of either of the preceding two paragraphs in the manufacture of clear solutions which give lacquers that will dry with a highly polished surface.

The invention also consists in the processes for the manufacture of cellulose aralkyl ethers substantially as herein described, and products including lacquers and the like when made by those processes, or by the obvious chemical equivalents thereof.

The following examples illustrate how the invention may be carried into effect, references to parts and percentages being to parts and percentages by weight:—

EXAMPLE 1.

A benzyl cellulose reaction mass, containing 5—40% benzyl chloride, but preferably 20% benzyl chloride, is treated in an autoclave with steam, at a pressure of 50 lbs. per square inch for one hour. The product is a creamy spongy mass, which is in the form of a soft paste, because of the thermoplasticity of benzyl cellulose. This mass hardens on cooling, and may be ground to a fine powder. It dissolves in mixtures of benzene spirits, xylene spirits and the like. The viscosity is reduced, and the solutions are clear.

GENERAL.

The treatment of benzyl cellulose or the like according to the present invention may be modified according to the nature of the benzyl cellulose or the like, or the degree of insolubility of that body, and according to the product desired. This treatment may also be employed to reduce the viscosity of the benzyl cellulose solution.

In the benzylation of cellulose a large excess of benzyl chloride is employed, and final traces of this liquid are difficult to remove. The presence of this benzyl chloride may, by the present invention,

be utilised to advantage. The benzyl cellulose reaction mass can be purified until it contains the requisite quantity of benzyl chloride, and the mass can then be treated with steam at increased pressures. The undesirable benzyl chloride is hydrolysed to benzyl alcohol, the benzyl cellulose is rendered completely soluble. This latter treatment of the reaction mass is valuable in the purification of benzyl cellulose.

The process therefore provides an improvement in the working up of the reaction mass, obtained in the manufacture of benzyl cellulose, being an improvement in the purification as well as a method of obtaining a soluble product. The time of heating and the steam pressure employed may be varied according to the amount of benzyl chloride present, and according to the viscosity or solubility of the benzyl cellulose desired.

The terms "soluble" and "insoluble" benzyl celluloses, or the like used above refer to benzyl celluloses which are soluble or insoluble in benzene spirits, toluene spirits and similar cheap solvents or mixtures of solvents.

The present invention includes benzyl celluloses and other cellulose aralkyl ethers in general, and in particular, special benzyl celluloses manufactured by the process described in co-pending British Application (Serial No. 327,714) No. 28,356/28.

Dated this 20th day of February, 1929.

MARKS & CLERK.

COMPLETE SPECIFICATION.

Improvements in and relating to the Manufacture of Cellulose Ethers.

We, IMPERIAL CHEMICAL INDUSTRIES LIMITED, a Company registered under the laws of Great Britain, of Imperial Chemical House, Millbank, London, S.W. 1 (formerly of Broadway Buildings, Westminster, London, S.W. 1), and DAVID TRAILL, a British subject, of "Cronulla", Ardrossan Road, Saltcoats, Ayrshire, Scotland, do hereby declare the nature of this invention and in what manner the same is to be performed, to be particularly described and ascertained in and by the following statement:—

This invention relates to cellulose derivatives, and more especially to aralkyl celluloses generally, for example benzyl cellulose, with reference to which latter body the invention will be more particularly described.

In investigations on benzyl cellulose, particularly in connection with lacquer production, we have found that the normal type of benzyl cellulose produced gives somewhat opalescent solutions, and lacquers made from the solutions dry with a matt surface. While such a surface might be utilised, an effort has been made to obtain water clear solutions which would dry giving a highly glossy surface. The cause of the opalescence has therefore been examined, and it appears to be due to the presence of ethers which are less degraded than the bulk of the benzyl cellulose.

The object of the present invention is to provide a method for lowering the viscosity of benzyl cellulose solutions and for the modification of benzyl cellulose, so that it will give clear solutions from which may be obtained lacquers that will dry with a highly polished surface, whilst at the same time retaining the advantage of the use of cheap solvents or mixtures of solvents, such as, for example, benzene spirits or toluene spirits.

The invention consists in a process for the solubilising of benzyl cellulose or other aralkyl cellulose which is wholly or partially insoluble in the usual organic solvents, which consists in treating the benzyl or other aralkyl cellulose with benzyl chloride or other aralkyl halide, and with water and steam at increased pressure.

The invention also consists in a process for the working up of a reaction mass such as may be obtained by the action of benzyl chloride on alkali cellulose, as described in our co-pending Application No. 327,714 (28,356/29), which comprises the purification of the reaction mass until it contains 3% to 25% of benzyl chloride or other aralkyl halide, and the conversion of the latter to benzyl alcohol.

The invention also consists in the application of products such as may be formed by the processes of either of the preceding two paragraphs in the manufacture of clear solutions which give lacquers that will dry with a highly polished surface.

The invention also consists in the processes for the treatment of cellulose

aralkyl ethers substantially as herein described, and products including lacquers and the like when made by those processes, or by the obvious chemical equivalents thereof.

The following examples illustrate how the invention may be carried into effect, references to parts and to percentages being to parts and percentages by weight:—

EXAMPLE 1.

A benzyl cellulose reaction mass, containing 5% to 40% benzyl chloride, but preferably 20% benzyl chloride, is treated in an autoclave with ten to twenty times its weight of water and with steam, at a pressure of 50 lbs. per square inch for one hour. The product is a creamy spongy mass, which is in the form of a soft paste, because of the thermoplasticity of benzyl cellulose. This mass hardens on cooling, and may be ground to a fine powder. It dissolves in mixtures of benzene spirits, xylene spirits, and the like. The viscosity is reduced, and the solutions are clear.

EXAMPLE 2.

A sample of benzyl cellulose which has been purified and is free from benzyl chloride, but which is not completely soluble in the usual solvents, is treated with ten times its weight of water containing 1–25%, but preferably 1–5%, of benzyl chloride for forty minutes in an autoclave, which is heated until the internal steam pressure is 40–50 lbs. per square inch. The benzyl cellulose is thus changed into a soluble form, which gives clear solutions of lower viscosity. In this way, for example, a viscosity of 10–100 c.g.s. units of a 10% solution of benzyl cellulose in a mixture of 80 parts by volume of toluene and 20 parts by volume of industrial spirits may be reduced to 0.1 to 10 c.g.s. units.

GENERAL

The treatment of benzyl cellulose or the like according to the present invention may be modified according to the nature of the benzyl cellulose or the like, or the degree of insolubility of that body, and according to the product desired. This treatment may also be employed to reduce the viscosity of the benzyl cellulose solution.

In the benzylation of cellulose a large excess of benzyl chloride is employed, and final traces of this liquid are difficult to remove. The presence of this benzyl chloride may by the present invention, be utilised to advantage. The benzyl cellulose reaction mass can be purified until it contains the requisite quantity of benzyl chloride, and the mass can then be treated with water and steam at in-

creased pressures. The undesirable benzyl chloride is apparently hydrolysed or partially hydrolysed to benzyl alcohol and condenses with the remaining benzyl chloride to form dibenzyl ether, and the benzyl cellulose is rendered completely soluble. This latter treatment of the reaction mass is valuable in the purification of benzyl cellulose, and in the suitable reduction of the viscosity.

The process therefore provides an improvement in the working up of the reaction mass, obtained in the manufacture of benzyl cellulose, being an improvement in the purification as well as a method of obtaining a soluble product of lower viscosity. The time of heating and the steam pressure employed may be varied according to the amount of benzyl chloride present, and according to the viscosity or solubility of the benzyl cellulose desired.

The terms "soluble" and "insoluble" benzyl celluloses, or the like, used above refer to benzyl celluloses which are soluble or insoluble in mixtures of 80 parts by volume of toluene, benzene, or xylene and 20 parts by volume of industrial spirits.

The present invention includes benzyl celluloses and other cellulose aralkyl ethers in general, and in particular, special benzyl celluloses manufactured by the process described in co-pending British Application No. 327,714 (28,356/28).

The modified aralkyl celluloses obtained have been found to have useful applications in various other directions, such as in moulding powders, safety glass, artificial silk, films, celluloid, floor covering, plastic wood and cable covering.

Having now particularly described and ascertained the nature of our said invention and in what manner the same is to be performed, we declare that what we claim is:—

1. A process for solubilising benzyl cellulose or other aralkyl cellulose, which is wholly or partially insoluble in the usual organic solvents, which consists in treating the benzyl or other aralkyl cellulose with benzyl chloride or other aralkyl halide, and with water and steam at increased pressure.

2. A process as claimed in Claim 1, for the working up of a reaction mass such as may be obtained by the action of benzyl chloride on alkali cellulose, as described in our co-pending Application No. 327,714 (28,356/28), which comprises the purification of the reaction mass until it contains 3% to 25% of benzyl chloride or other aralkyl halide, and the conversion of the latter to benzyl

alcohol.

3. The process which consists in the application of products formed by the processes of either Claim 1 or Claim 2, in the manufacture of clear solutions which give lacquers that will dry with a highly polished surface.

4. Processes for the treatment of cellu-

lose aralkyl ethers substantially as herein described.

5. Products including lacquers and the like when made by the processes described or claimed herein, or by the obvious chemical equivalents thereof.

Dated this 18th day of November, 1929.

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10

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