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TABLETED POLYCHLOROPHENOL SALTS

Kent R. Fox, St. Louis, and Ira Hatfield, Webster Groves, Mo., assignors to Monsanto Chemical Company, St. Louis, Mo., a corporation of Delaware

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The present invention relates to water-soluble polychlorophenol salts, particularly pentachlorophenol salts, and refers to a new and improved physical form of such salts which permits of their more advantageous handling in commerce and in industry without substantial impairment of their ease of solubility.

Pentachlorophenol and tetrachlorophenol salts in the dry powdered condition or in the form of their monohydrates, particularly the sodium salts of these phenols, are very light in bulk and are easily blown about. In handling such materials it is difficult to avoid dusting. These salts produce local irritation of the skin and mucous membranes of the nose and throat and on continued exposure may produce a dermatosis of the skin characterized either by redness or an acneform eruption. The salts even when inhaled in small amounts produce sneezing followed by excessive rhinorrhea, that is, excessive discharge of mucous from the nostrils. Such materials, therefore, are hazardous and have been avoided because of the special precautions required in their use.

Sodium pentachlorophenate or the monohydrate in the dry powdered condition readily dissolves in water by stirring. However, when it is merely wetted with sufficient water, hydrate formation occurs and the resulting hydrated compound sets to a mass resembling plaster of Paris which is far more difficult to dissolve. Although the hydrated salt is less hazardous to handle, it is surprisingly more difficult to use because of its failure to dissolve rapidly. Furthermore, dry sodium pentachlorophenate which has been exposed to the atmosphere will over a period of time develop varying proportions of water-insoluble components. The insoluble material represents a loss to the consumer.

The primary object of the present invention is to produce water-soluble salts of polychlorophenols in a form such that they are readily and completely soluble in water, are non-dusting, are not appreciably affected by exposure to the atmosphere, and which dissolve quickly and completely even after storage.

We have found that the above objects are attained in an especially advantageous manner by producing the polychlorophenol salts in the form of tablets or briquettes in the manner described hereinafter.

The difficulty in attaining the objects of the present invention has been encountered in making a compacted mass which while in compact form should not be fragile and affected by atmospheric moisture and which, on the other hand, should when dropped into water dissolve rapidly therein. Excellent tablets or briquettes can by produced by simple moistening of sodium pentachlorophenate with water and molding, but the resulting tablets resemble set plaster of Paris, are difficultly soluble, and are therefore entirely unsatisfactory. Anhydrous sodium pentachlorophenate itself can be compressed into a tablet or briquette but the mass crumbles on standing. Any binding agents containing water which are added to aid in tableting or briquetting, for example, sodium silicate solutions, produce hydration of the salt, whereas bituminous, tarry or similar binding agents are not usable because they, of themselves, being insoluble, make the resulting tablet or briquette extremely difficult to dissolve. Furthermore, when sodium silicate is used as a binding agent for the tablets or briquettes, it produces a powder which adheres to the metal equipment and the resulting tablets are not readily disengaged from the mold after compression. On the other hand, when starch is added to the material as a binding agent it yields a tablet of more or less lamellar form, as a result of which it crumbles and disintegrates readily. The application of the common methods heretofore known, failed to produce coherent masses of required solubility in water.

The present invention is based upon our unexpected discovery that sodium tetrachlorophenates and pentachlorophenate of controlled moisture content will form into coherent tablets or briquettes when compressed in a particular manner, hereinafter described in full, to produce the desired objects set forth above. In such tablets or briquettes the outer surfaces are in more or less coherent form whereas the interior is in particulate or granular form. The tablet is impervious to atmospheric moisture and air to a remarkable degree and the salt in the interior of the tablet or briquette is thereby protected from decomposition and dehydration. The tablets, briquettes or cakes are substantially non-crumbling, non-hydroscopic and non-deliquescent, that is, they neither absorb nor liberate water to any appreciable extent. The small content of moisture required for producing such coherent tablets or briquettes is without substantial effect on the solubility in water of the formed mass. When the salt is thus used in the form of coherent tablets there is little danger of the occurrence of dusting. Even should any small particles become disengaged from a formed cake or tablet by abrasion or dropping, they are in a

compressed or agglomerated form and not in the form of a fine bulky powder that is a hazard.

In an example of the method of practicing the invention a powdered sodium pentachlorophenate containing from approximately 3% to 10% of moisture or water is used. Such moisture or water is probably held in the salt as water of crystallization, inasmuch as the salt itself is substantially dry. This water content may be controlled in the production of the salt or water may be incorporated into dried sodium pentachlorophenate powder of a lower moisture content by careful uniform mixing in the customary manner, care being taken to avoid any local concentration of water which would result in hydration to the less soluble form. By controlling the drying operation used in the production of the salt this moisture content can be maintained most conveniently. This optimum moisture content is variable to some extent, the limits being approximately 3% to about 20%, which corresponds to from roughly ½ to 4 molecules of water for each molecule of anhydrous sodium pentachlorophenate. At about a water content of 30% the product produces a hydrate which sets similar to plaster of Paris. With proportions of water from about 30% to as high as 70%, the products are solids. Products having more than 70% or thereabouts of water are fluid suspensions or solutions, for example, 25 parts of dry sodium pentachlorophenate in about 75 parts of water yields a clear solution at room temperature. For best results in tableting or briquetting, a water content of about 3% to 10% is most satisfactory. Smaller water contents require greater pressure in the tableting operation and yield tablets which disintegrate too readily. Products of higher water content (containing from about 10% to 20% water) do not compress with any substantially greater ease than those of about 10% water content, and since these products have a higher water content, the sodium pentachlorophenate becomes more expensive to ship and the tablets or briquettes themselves have a smaller margin of safety with regard to any detrimental effects which might result from any extraordinary exposure to moisture or accidental wetting.

The powdered sodium pentachlorophenate of controlled moisture content is tableted or briquetted in the following manner: The powder is fed to a tableting machine of conventional design in which the pressure applied to the mold is from approximately 5,000 to 20,000 pounds per square inch. The resulting molded mass, which at this stage is easily broken when dropped, is then disintegrated or granulated in a cob mill or a suitable grinding machine to yield particles of about 1 to 10 mesh in size, the preferred size being approximately 4 mesh. The particulate compressed material or granulations are then subjected to a second compression under approximately the same conditions in a mold.

The mold in this first compression can be of any shape or size but the mold used in the final compression (recompression) should be that corresponding to the form in which the final tablets are desired. After this second compression the resulting tablet is firmly coherent and will easily resist abrasion or breaking during handling and transportation. The recompressed tablet is not deliquescent or efflorescent and is substantially impervious to atmospheric moisture or carbon dioxide. On standing exposed to the atmosphere for long periods there is no substantial disintegration of formation of water-insoluble compounds. To prepare aqueous solutions of the salts from the tablets, agitation is desirable, preferably agitation by means of a jet of steam through the solution.

Instead of using one preliminary compression and granulation as above, the salts may with advantage be subjected to two or more preliminary compressions to produce compacted particulate masses or granulations. The preliminary and final compressions may be conducted if desired on suitable rotary briquetting machines, although in this event more than two compressions and granulations are usually necessary since such briquetting machines permit the application of great pressures but do not in any one compression reduce the bulk of the material sufficiently.

The pressures which are applied to the mold in the tableting operation are somewhat variable, but are within a preferred range of from about 5,000 to 20,000 pounds per square inch. Less pressure may be used in the preliminary molding as can be well understood. The pressures to be applied will to a large extent vary with the degree of hydration of the salt within the range specified and its compactness. If more than two compressions are resorted to somewhat smaller pressures may be used in each compression.

The tablets or briquettes of the present invention are intended particularly for the preparation of baths for the treatment of wood to prevent the growth of sap-staining organisms and other fungous growths. In such use a precipitation of the polychlorophenol appears in the bath after prolonged wood treatment. This precipitation in such baths can be prevented and the quality of the tablet or briquette is furthermore markedly improved by the incorporation with the salt of a small proportion of sodium hydroxide or potassium hydroxide or other suitable strong alkali. Proportions of from 1% to 10% of sodium hydroxide have an especially advantageous effect on the quality of the resulting tablet and aqueous baths made with such sodium-hydroxide-containing tablets can be used for long periods without any difficulties from precipitation of the free chlorophenols. The sodium hydroxide can be advantageously incorporated in the original preparation of the salt, that is, prior to the drying of the salt. Tablets containing alkali in these proportions, surprisingly, are not affected to any substantial extent by prolonged exposure to the atmosphere, any more than tablets not containing alkali.

To aid in solubilizing the tablet or briquette it is desirable also to add a small proportion of a wetting agent such as a sulfonated high-molecular-weight alcohol or preferably a sulfonated alkylated aromatic hydrocarbon such as that known to the industry under the trade name "Santomerse". For this purpose less than 1% by weight of "Santomerse" is ample.

Additions of coloring agents, substances which exert a synergistic action upon the polychlorophenol, talc or other mold lubricants, and similar components of a secondary nature in small proportions may be made prior to the compression without necessitating changes in the molding procedure.

The invention is particularly applicable to the tableting or briquetting of sodium tetrachlorophenates and pentachlorophenate. The optimum moisture content of the respective salts or mixtures of salts to be used in the tableting will be

influenced to some extent by the factors pointed out hereinabove but will be within the range of 3% to 20% which has been specified for sodium pentachlorophenate.

Inasmuch as this specification comprises preferred embodiments of the invention it is to be understood that it is not limited to these specific examples and that variations and modifications can be made in accordance with the principles disclosed herein without departing substantially from the invention, which is defined in the appended claims.

The term "polychlorophenols" as used in the description refers to the tetra and penta-chloro-monohydroxy-benzenes. Tetrachlorophenol, as used in the specification and the claims, is to be understood to mean any of the isomeric tetrachlorophenols or mixtures thereof. It is to be understood that mixtures of sodium tetrachlorophenates and sodium pentachlorophenate are also contemplated.

What we claim is:

1. A formed compressed coherent mass consisting predominantly of sodium pentachlorophenate having a moisture content of from 3% to 20% and containing from 1% to 10% of sodium hydroxide, and being characterized by being readily and substantially completely soluble in water, said product being obtained by compression of the powdered salt, subsequent granulation of the compacted mass, and final molding thereof.

2. A formed compressed coherent mass consisting predominantly of a sodium salt selected from the group consisting of the sodium salts of tetrachlorophenol and pentachlorophenol having a moisture content of from approximately 3% to 20% and containing from 1% to 10% of sodium hydroxide, said tablet being characterized by being readily and substantially completely soluble in water.

3. A formed compressed coherent mass consisting predominantly of sodium pentachlorophenate having a moisture content of from approximately 3% to 20% and being readily and substantially completely soluble in water.

4. A recompressed tablet consisting predominantly of a sodium salt selected from the group consisting of the sodium salts of tetrachlorophenol and pentachlorophenol having a moisture content of from approximately 3% to 20% and containing from 1% to 10% of sodium hydroxide, said tablet being readily and substantially completely soluble in water.

5. A recompressed tablet consisting predominantly of a sodium pentachlorophenate having a moisture content of from approximately 3% to 20% and containing from approximately 1% to 10% of sodium hydroxide, said tablet being characterized by its resistance to breakage and abrasion and by being readily and substantially completely soluble in water.

6. The method of tableting a salt selected from the group consisting of the sodium salts of tetrachlorophenol and pentachlorophenol, consisting in incorporating uniformly with said salt a proportion of water such that the water content of the salt corresponds to from 3% to 20% of the mass and a proportion of sodium hydroxide such that the mass contains from 1% to 10% thereof, and thereafter by means of compression and disintegration producing a mass of said salt of reduced bulk in particulate granular form, and subsequently subjecting said granulated mass to a final molding to produce a coherent tablet which is readily and substantially completely dissolved in water.

KENT R. FOX.
IRA HATFIELD.