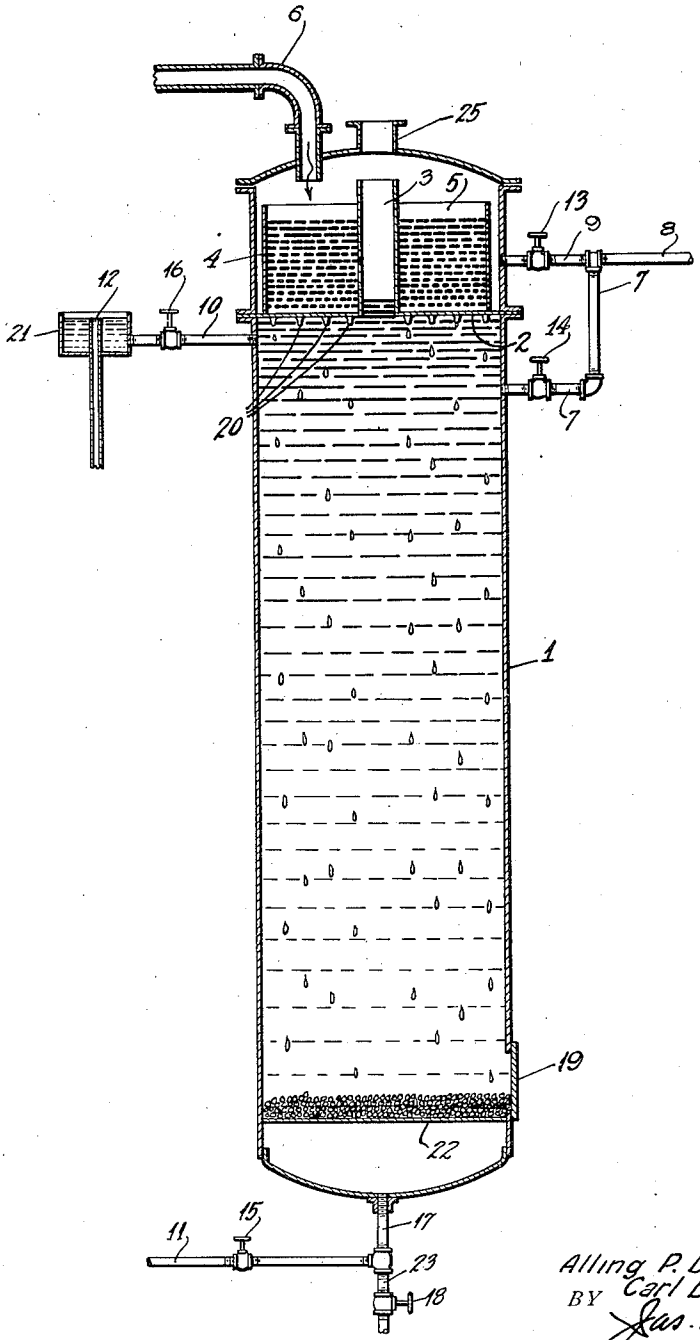
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DINITROBENZENE PELLET

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DINITROBENZENE PELLET.

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Dinitrobenzene has hitherto been placed on the market in the form of large blocks or lumps, or in the form of a rough granular material of irregular shape, size and fineness, which materials, when dry, generally include a large amount of dust. Dinitrobenzene is highly poisonous, and either its vapor or its dust, when inhaled to any appreciable extent, will seriously affect workers employed in the manufacture or use thereof. Poisoning can readily be contracted by handling such material, containing fine particles and dust, and in breaking up large lumps for charging into vessels.

To minimize the harmful effects to workmen, when handling such material, it has frequently been the practice to handle and ship dinitrobenzene in moist form. That is to say, during its process of manufacture, it is made to contain sufficient water to minimize dusting. The use of water will unquestionably lessen the danger to workmen, but if the material is left exposed for any appreciable time, the aqueous content will evaporate and dusting will occur, resulting in the injurious effects referred to.

Moreover, when water is embodied in the commercial material it is usually not uniformly distributed and it is difficult to obtain a representative sample for the determination of moisture. It is therefore difficult to know how much actual dinitrobenzene is contained in a given weight of the moist material.

With these considerations in mind, the object of the present invention is to provide dinitrobenzene in convenient commercial form, in order to permit it to be shipped and handled in a substantially dry state, without appreciable dusting, in such manner as not to render the workmen liable to poisoning.

Another object of the invention is to provide a simple, economical and efficient method whereby a new commercial form of material can be produced.

Speaking generally, the material of this invention consists of dinitrobenzene in pellet form, having a very close and marked resemblance to the small, smooth pebbles found in the beds of streams and in gravel pits. Because of its form, we may figuratively refer to the material as dinitrobenzene pebbles.

A characteristic feature of the material consists of the glazed surface of the pebbles which, in practice, may be made of a substantially uniform size, and with rounded or curvilinear exteriors, presenting no sharp points or edges and having a substantially uninterrupted smooth and glazed exterior. The glazed exterior and unbroken rounded surfaces effectually eliminate dusting of the material which would otherwise result from friction between them when they are handled in bulk. The rounded form of the material presents less surface than irregularly shaped grains of the same weight will have, and consequently less material will volatilize from the surface.

The material in the pebble form described can be practically and safely handled and shipped in a substantially dry condition, without being open to the well recognized disadvantages of the commercial product now on the market.

In practically carrying out the method of this invention in its preferred form, the dinitrobenzene is subjected to a temperature above its melting point, and after being thereby transformed into a liquid, is passed through a progression of successively lower temperatures of a cooling fluid, for example, water. The drops or globules of dinitrobenzene are formed in water, the temperature of which is at or above the melting point of the dinitrobenzene, and thereafter such drops or globules are caused to gravitate through successively cooler regions whereby they are solidified and formed into the pebble-like pellets desired. Having been thus formed and solidified, the pebbles are removed from the water in which they were formed, and are dried, whereupon they are ready for shipment.

Features of the invention, other than those

adverted to, will be apparent from the hereinafter detailed description and claims, when read in conjunction with the accompanying drawings.

The accompanying drawing illustrates, in a diagrammatic manner, apparatus for carrying out the method of this invention but the showing therein made is to be understood as illustrative only, and not as defining the limits of the invention. In the diagrammatic showing, the apparatus is shown in central section.

Referring to the drawings, 1 designates a relatively deep tank, provided near its top with a transverse partition 2, upwardly from the center of which extends a pipe 3, which extends as high as the annular wall 4, and is open at the top.

Also extending upwardly from the partition 2, and adjacent to the lateral wall of the tank is an annular wall 4, which is coaxial with the pipe 3, and forms therewith an annular vessel 5. A pipe 6 leads through the top of the tank, and through this pipe the material to be treated is adapted to be discharged into vessel 5. The wall 4 is spaced from the side wall of the tank, and one branch 9 of a steam supply pipe 8 leads into the space thus provided. A second branch 7 of the steam supply pipe leads into the tank below the partition 2. Also leading into the tank below the partition 2 is a pipe 10, to which is connected a box 21. Within the box 21, is an overflow pipe 12, which serves to maintain the level of the water in the tank 1 approximately at the partition 2. The branch pipes 9 and 7 are provided with valves 13 and 14 respectively, and the pipe 10 with valve 16. Leading into the tank at or near the bottom is a pipe 17 to which is connected a cold water feed pipe 11, controlled by a valve 15. Also connected to pipe 17 is a drain pipe 23 controlled by a valve 18. At one side of the tank 1, near the bottom, is a manhole 19, normally sealed. In practice this is preferably positioned just above the partition 22.

In the lower part of tank 1, preferably close to the bottom is a partition 22. This partition is provided with openings and, if desired, is covered with a cloth whose weave is suited to prevent the passage of pellets of dinitrobenzene, while permitting the passage of water. In the cover of tank 1 is a vent 25, which may be open to the atmosphere.

A portion of the partition forming the bottom of vessel 5 is provided with a number of nozzles 20, so formed and shaped as to function as droppers, so that material contained within the vessel may be discharged by gravity through the nozzles 20 in the form of drops or globules.

In practice, the parts shown in the drawing may be made of any appropriate size, and may be shaped differently or differently arranged from the specific showing made, but the construction illustrated diagrammatically shows a simple form of apparatus by which the method of this invention may be conveniently and economically carried out. The specific method will now be described.

The tank 1 is first filled with a liquid of less specific gravity than liquid dinitrobenzene. In practice, this liquid may vary, but we have found that water will give satisfactory results. This water is introduced into the apparatus of the drawing by closing the valve 18 and opening the valve 15, so that the water flows through pipes 11 and 17 into the tank. The tank is filled till the water overflows through pipe 10, box 21, and pipe 12. The valve 15 is then closed.

The valve 14 is then opened to permit the entrance of steam into the tank, for the purpose of heating the water or other liquid in the upper portion of the tank to a temperature at least as high as the melting point of dinitrobenzene. After the desired temperature is attained, the valve 14 is closed. During this heating, excess water formed by the condensation of steam can escape by pipe 10, box 21, and the pipe 12.

In practice the water at the top of the tank will of course heat first. That is to say, the upper portion of the water will be the hottest, while the water at the bottom of the tank will be substantially cold. Between the partition 2 and the bottom of the tank, the water will be of progressively lower temperature from the partition downward, and this condition may be figuratively referred to as a succession of progressively cooler regions in a downward direction.

After the water in the vicinity of the nozzles has reached the desired temperature, dinitrobenzene, at a temperature to maintain it liquid, is fed through the pipe 6 into the vessel 5 and immediately starts to drip through the nozzles 20 in the form of drops or globules. Steam is turned on at the valve 13 and this steam circulates about the wall 4 and in the head of the tank above the vessel 5, maintaining the dinitrobenzene in liquid form so that it may continue to drip through the nozzles.

Melted dinitrobenzene is of greater specific gravity than water, and will thus pass down through the nozzles slowly and form at the tips of the nozzles into drops, which, after being formed, will free themselves from the nozzles and drop through the water to the partition 22. It is of course essential to have the water in the vicinity of the nozzles sufficiently hot to preclude any tendency for the material to solidify at the nozzles, and the temperature of the water is therefore kept sufficiently high to permit unrestricted formation of drops of substantially uniform size by turning on steam

through valve 14. Should the temperature of the water near partition 2 be higher than desired, or should the depth of the region of hot water in the top of tank 1 be deeper than desired, cold water may be admitted through pipes 11 and 17, the displaced water overflowing through pipe 10, box 21 and pipe 12.

As the drops leave the nozzles, and gravitate downward, they slowly pass through successively cooler regions of water, whereby they are cooled and caused to solidify, the tank being sufficiently high to permit the solidification of the globules by the time they reach the partition 22. They collect on this partition as the process continues until a sufficient quantity is obtained, or until they have built up to such a height as to unduly decrease the distance between the nozzles and the accumulated product. At this point, steam is turned off at valve 14, if in use, and the hot water in the top of tank 1 is caused to overflow through pipe 12, by admitting cold water through pipes 11 and 17. If desired, the regions of hot water may be drawn off by a valve suitably located in the side of tank 1, said valve being not shown in the drawing, but located just below the point where the water is of a temperature which it would be undesirable to communicate to the accumulated pellets.

The drain valve 18 is then opened and the water in tank 1 allowed to run out through the partition 22. If the hot water previously in the top of tank 1 had not been first removed, as just described, it would finally reach the accumulated pebbles, as the water drained out through valve 18, and sinter them or melt them. When the water has been drained down below the level of partition 22, manhole 19 is removed, and the pebbles are taken out and either artificially dried or permitted to dry in the air. When the material is dried down to a moisture content of about one or two percent, it is in commercial form, as it is then approximately dry.

The finished product is found to be in the form of pellets, which closely resemble small pebbles, and are therefore figuratively described as dinitrobenzene pebbles. The individual pebbles have a glossy surface, and the contour of such pebbles is smooth and curvilinear. There are no edges or corners, and the product of the operation is substantially uniform in size. The size of the pebbles is of course dependent upon the size of the drops formed at the nozzles. By controlling the head of liquid material above the nozzles and the specific gravity of the liquid in tank 1, and by suitable design of the nozzle, the size of the finished pellets can be controlled within certain limits.

It will of course be understood that the specific gravity of the liquid contained in the tank below partition 2 will have an effect upon the size of the pebbles, in that the nearer the specific gravity of the liquid is to that of the liquid dinitrobenzene, the larger will be the pebbles, other factors such as temperature, liquid head and nozzle size being constant. We have operated the apparatus with water, whose specific gravity had been raised by addition of other substances, with satisfactory results. Liquids other than water or water solutions may also be used. We have found however that water is highly practical and gives thoroughly satisfactory results, aside from being cheap and convenient.

We wish to particularly call attention at this point to the smooth glazed surface found on the pellets or pebbles made in accordance with the process which we have described. It is this feature which permits the handling of the material in bulk without incidental dusting, and it is this feature also which minimizes volatilization of the material, when in the substantially dry condition to which we have referred.

Of pronounced practical importance moreover is the fact that the material is converted by the method of this invention into a more satisfactory commercial form, by a process which does not expose the workmen engaged in the carrying out of the process to the dangers heretofore often prevalent in the handling of dinitrobenzene, such as breaking up and handling blocks of the solid material. The material passes into the apparatus through a pipe 6 and is entirely enclosed during the carrying out of the process in a closed chamber. When removed through the manhole, the pellets have acquired the smooth hard surface to which we have referred. The production of such material can be carried on with absolute safety to the workmen, as fumes and dusting do not accompany the practising of the method, as is the case with the usual method of casting the material in molds and breaking it up.

The foregoing detailed description deals with the invention in its preferred practical form, but the invention is to be understood as fully commensurate with the appended claims.

The term dinitrobenzene, as used in the foregoing specifications, is understood to mean the commercial product usually described by this term, and which consists in a mixture of meta, ortho and para isomers.

Having thus fully described the invention, what we claim as new and desire to secure by Letters Patent is:

1. As a new article of manufacture, dinitrobenzene pebbles having smooth glazed surfaces.

2. As a new article of manufacture, dini-

trobenzene pebbles of smooth curvilinear contour and having glazed surfaces.

3. As a new article of manufacture, dinitrobenzene pebbles of approximately uniform size, each of which pebbles has a smooth glazed surface.

4. As a new article of manufacture, dinitrobenzene pebbles having smooth glazed surfaces and of not exceeding two percent moisture content.

In testimony whereof we have signed the foregoing specification.

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