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GRANULATION METHOD

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Fig. 1

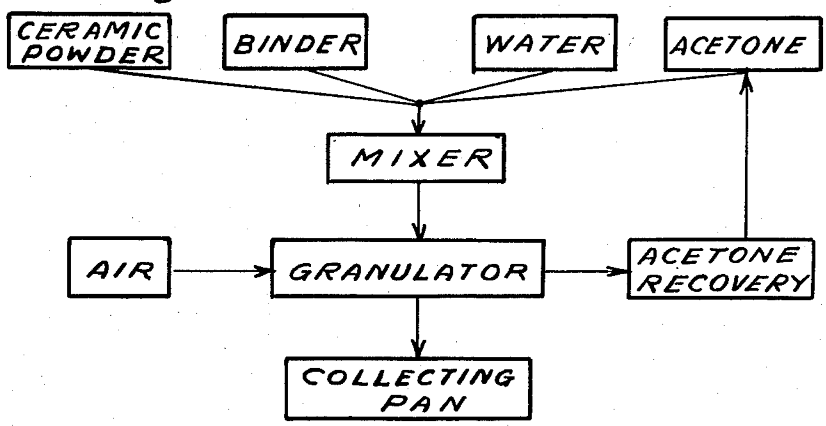
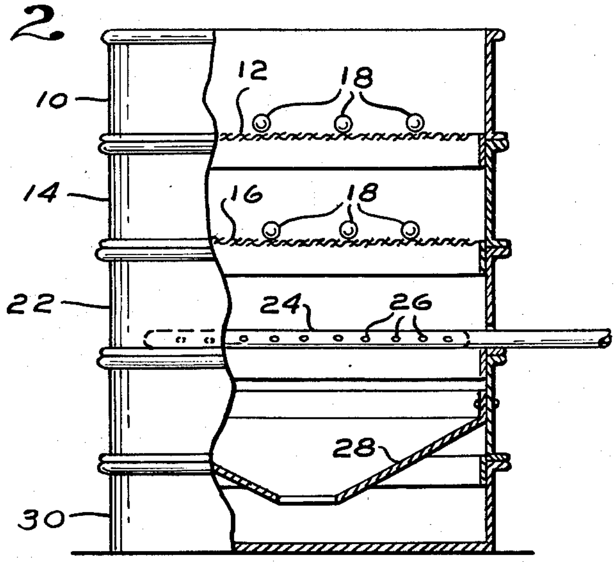


Fig. 2



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GRANULATION METHOD

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This invention relates to the granulation of finely divided materials, particularly ceramic materials, preparatory to molding into pellet, tablet, or other form for direct use or for subsequent processing, such as sintering.

It is highly desirable, especially in a continuous molding operation, to produce molded bodies that are physically homogeneous and have a consistent density. This is particularly true where, as in the case of ceramics, the molded bodies are subsequently consolidated or densified by firing at an elevated temperature. Otherwise the shrinkage which normally occurs during such firing is uneven thus resulting in a deformed, strained, or otherwise defective product.

To this end, it is important to charge a controlled amount of material into the pressing die, and then to distribute the applied pressure evenly throughout the mass. This requires free-flowing material, a small liquid content for lubricating purposes, and a uniform bulk density. The latter is largely a matter of particle size control so that segregation of the material does not occur during handling of the batch or charging of the pressing dies.

The optimum size range for any particular material or type of product will vary of course. By way of example, however, it is generally desirable, in pressing certain types of ceramic pellets, to employ granulations that are substantially within the range of 35 to 200 mesh. Reference to mesh size is in accordance with common usage in the art and, in the present instance for example, means that the granulations are of such a size that they will pass through a standard 35 mesh screen but are large enough to be retained on a 200 mesh screen.

It is common practice then to granulate finely divided or powdered materials before molding. A variety of granulating machines and mechanical methods have been proposed. In general these involve subdividing a moistened mass of powdered material. One conventional type of machine includes means for rapidly chopping or cutting the moistened mass and collecting it as it passes through a perforated plate or screen. Another type employs a series of rotating bars or rollers to force the wetted material through a series of screens of decreasing size.

Various problems have however been encountered in the operation of such granulating equipment. One of the most series arises from the need for critical control of the liquid content in the material fed to the granulator. Thus it has been found that a slight excess of liquid markedly slows down the process and tends to blind or smear over the screens or perforated plates through which the granulated material must pass. On the other hand, too little liquid results in an excessive amount of powdered or ungranulated particles being formed. A further undesirable feature has been the tendency to form a considerable amount of fine material even under ideal operating conditions. As a result ceramic materials granulated by such methods may be expected to contain as much as 25% or more of material smaller than 200

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mesh size, commonly referred to as —200 mesh material.

It is possible, of course, to screen this material and, by selecting proper amounts of various sizes, produce a desired mixture. While this expedient is frequently resorted to, it introduces the undesirable feature of either discarding a substantial amount of the granulated material or returning it for regranulation, either of which is uneconomical.

It has also been proposed to produce granulated material by a process known as spray drying. While this has been successfully used in many instances, it involves heating the material to elevated temperatures which is quite undesirable where the material is easily oxidized or otherwise chemically active.

In general prior granulating machines and non-manual methods have been designed for large volume operations in connection with inexpensive raw materials such as clays where small material losses in the course of the operation are not particularly significant. Such equipment and methods have not however proven readily adaptable to smaller scale operations, and particularly to the handling of expensive raw materials such as uranium oxide where even a small loss is economically important.

It is then a primary purpose of this invention to provide an improved method of granulating finely divided materials, particularly ceramic materials. A more specific purpose is to provide such a method which is capable of producing a high yield of granulated material with a size distribution well adapted to pressing. A further purpose is to provide a method of producing such granulations with a suitable moisture content for pressing. Another purpose is to provide a flexible granulating method particularly adapted to the handling of small amounts of expensive material, but also capable of larger volume production as well. A still further purpose is to provide a method of granulating materials which are readily oxidizable or otherwise chemically active. Still another purpose is to provide a method which may be performed by modifying conventional granulating machines.

The invention and a manner in which it may be practiced are hereafter described in conjunction with the accompanying drawing wherein,

Fig. 1 is a flow diagram illustrating the steps involved in the method and,

Fig. 2 is an elevational view partly in section schematically illustrating a granulating system in accordance with the invention.

The invention provides a method of granulating a finely divided material which comprises mixing the material with two liquids having substantially different rates of volatilization to form a plastic mass, and removing the more volatile liquid while subdividing the mass into granules smaller than a selected mesh size. In a preferred form, the invention involves subdividing the mass into granules capable of passing through a perforated partition member while simultaneously exposing the mass to a current of air whereby the more volatile liquid is substantially removed and granulation of the material is facilitated.

The method is particularly applicable in granulating a ceramic powder, such as uranium oxide or alumina, for subsequent pressing and firing to a dense body and is so illustrated in the flow diagram of Fig. 1. As there indicated, the ceramic material is combined and thoroughly mixed with a binder and two liquids, such as water and acetone, to form a consolidated mass. The binder, added to impart strength after molding, is not a feature of the present invention, and any compatible binder may be selected.

An advantage of the present method is that the total amount of fluid present is not especially critical and may be varied depending on the type of mixing and granulating apparatus used as well as the material itself. For present purposes it is sufficient to incorporate only enough liquid to form a pasty, consolidated mass which can be cut into coarse chunks for subsequent granulation, this being easily determined once operating conditions are specified. However, if the material is to be initially wet-milled for example, a larger amount of fluid may be added to form a slurry. Subsequently the slurry must be dried to a pasty or plastic condition for cutting into chunks or otherwise forming coarse granulations.

A primary feature of the present invention is that a mixture of two liquids, having widely different rates of volatilization or vapor pressures at constant temperature, is employed in wetting the ceramic powder. The specific example of water and acetone, chosen for illustration, is generally suitable, and illustrates the desired difference in volatilization rates. Other similar mixtures might be used as well in accordance with the principles set forth below. In particular other compatible and relatively volatile organic liquids such as simple alcohols, ethers and hydrocarbons may be used with water.

We have found that when a mixture of this nature is exposed to a current of dry air the more volatile liquid, e.g. acetone, is rapidly, and substantially completely, removed while only a small fraction of the less volatile liquid, water, is removed. We have further found that each ceramic material has an optimum moisture content for good pressing in the granulated state. This varies with the material but is generally on the order of 1–3% and may easily be determined for any given material and process.

This then permits addition of only slightly more water than the amount desired in the final granulated product, say up to a third more depending on the relative volatilization rates, and a relatively much larger proportion of volatile liquid in mixing the ceramic powder. In the course of granulating, substantially all of the more volatile liquid may be removed while only the slight excess of water is removed and a closely controlled, optimum amount for proper pressing is retained.

In a preferred embodiment of the invention then, a finely divided material is prepared for granulation by incorporating in the material substantially the amount of moisture or other wetting fluid desired in the granulated product and a relatively larger amount of a more volatile liquid, the total being at least sufficient to form a plastic, consolidated mass. Thereafter substantially all of the more volatile liquid is removed during granulating, leaving the desired amount of moisture in the granules.

We have further found that continuous drying of the coarse granules, particularly their surfaces, while they are worked through screens of decreasing size, provides rapid reduction in size without blinding or smearing of the screens. Furthermore, the amount of fine material, that is material under 200 mesh, is relatively small. It is not certain whether the tendency to form such fines is actually diminished or whether the fines still form and quickly reaggregate. It is our belief that the latter occurs to some extent at least and is facilitated by imparting lateral motion to the granulated material.

Turning again to the drawings then, the plastic mixture of liquid, binder and ceramic powder is coarsely granulated by cutting, beating, milling or other conventional means. With small batches a convenient method involves working the mixture through a ten mesh screen. The coarse granules thus formed are then introduced into a fine granulating system, such as that illustrated in Fig. 2, for forming 35 to 200 mesh material, and the fine granules collected.

The granulating system of Fig. 2 includes a conventional sieve **10** having a standard twenty mesh screen **12** mounted above a second sieve **14** having a thirty-five mesh screen **16**. Balls **18** are provided to assist in working the material through the screens. Sieves **10** and **14** are in turn mounted over a spacer **22** having a perforated circular coil **24** mounted on its inside wall and extending out to a flexible connection (not shown). Air under controlled low pressure is introduced through perforations **26** of coil **24** and flows upwardly or counter-current to the descending material being granulated. The granules formed in this manner as collected on a collecting cone **28** and funneled into pan **30**.

While imparting of any type of shaking or vibratory motion to the assembly is capable of performing the granulating process, we prefer to impart an eccentric rotary motion in a lateral direction by mounting the assembly on a shaking apparatus designed to impart such motion, a suitable apparatus being one sold by the Taylor Mfg. Co. under the name Ro-Tap Shaker. With this type of motion the granulating time is reduced and an optimum opportunity for reaggregation of fines is provided.

The apparatus as shown is particularly designed for operation on a laboratory scale with small lots of material. The principles involved, however, may readily be embodied in larger scale commercial equipment. Thus commercial machines, earlier referred to as embodying means for rapidly cutting a moistened mass into granules to pass through a perforated partition, or means for mechanically working the mass through a series of screens, may be modified to perform the present method. Such machines need be modified only to the extent of adding a source of counter current air through their screens or partitions. Tests have shown that the problem of screen blinding is greatly minimized and a higher yield of properly sized granulations is then obtained on such equipment.

By way of further illustrating the invention, reference is made to the following specific examples,

Example I

A solution of 6 cc. of water in 24 cc. of acetone was added to 300 gms. of ballmilled uranium oxide. UO_2. An addition of one weight percent of a polyethylene glycol type binder was made to the UO_2 prior to ballmilling this material. The solution and UO_2 were mixed until the powder was thoroughly wetted. The wetted material was forced through a 10-mesh sieve with a spatula, and the resulting coarse granules introduced at the top of an assembly, such as illustrated in Fig. 2, mounted on a sieve shaker. The assembly consisted, in descending order, of a 20-mesh sieve, a 35-mesh sieve, a spacer containing a copper air-input loop, a collection cone and collecting pan. Two refractory balls were placed on each sieve to aid in working the granules through the sieves. A counter-current air flow on the order of 1000 cc./minute was maintained to volatilize the acetone. After four minutes of shaking, the resultant granules, collected in the pan, were dry (approximately 1.3% moisture which is beneficial to pressing), free flowing, soft, and had a good size-distribution for pressing. A typical screen analysis follows:

Screen:	Weight percent
−35+48	45.0
−48+65	26.6
−65+100	12.1
−100+150	7.9
−150+200	3.4
−200+325	3.0
−325	2.0

Example II

Ten pounds of alumina powder having an average size of 900 mesh was manually mixed with a solution composed of 2.3 pounds acetone, 0.3 pound of water and 0.1 pound of a polyethylene glycol type binder. The mixture was cut into coarse chunks and rolled through a ten mesh size screen. The coarse granules thus formed were introduced in half pound lots over a granulating assembly as in Example I for a period of about five minutes per lot with a low pressure, that is under five pounds pressure, air current flowing. The material collected had a moisture content of 2.60% and a typical screen analysis as follows:

Screen:	Weight percent
—35+48	37.7
—48+65	24.6
—65+100	17.9
—100+150	8.3
—150+250	6.0
—250	5.1

Example III

A mixture of coarse granulated alumina prepared in accordance with Example II was treated in identical manner except that the air current was varied slightly so as to employ a seven minute granulating period per lot. The material collected had a moisture content of 2.08% and a screen analysis as follows:

Screen:	Weight percent
—35+48	49.7
—48+65	24.9
—65+100	14.3
—100+150	6.4
—150+250	3.7
—250	1.1

Example IV

A 150 gram batch composed of 78.6% Al_2O_3 and 21.4% UO_2 by weight was mechanically mixed with 3 cc. H_2O, 30 cc. acetone and 2% by weight of a polyethylene glycol binder to form a consolidated mass in which the acetone content was reduced by evaporation to about 12 cc., thus providing a proper consistency for coarse granulation. This was coarsely granulated, introduced over a screen granulator as in Example I and exposed to a counter current of about 1300 cc./minute of air for a period of 3 minutes. The screen analysis was as follows:

Screen:	Weight percent
48	0.9
65	38.0
100	24.8
150	16.8
200	8.5
325	9.1
—325	1.9

Example V

A consolidated mass was prepared as in Example IV from a mixture by weight of 83.2% ZrO_2 and 16.8% UO_2. The material was granulated under the same conditions and produced the following screen analysis:

Screen:	Weight percent
48	38.6
65	23.5
100	11.9
150	7.5
200	3.8
325	5.1
—325	9.7

It will be seen then that the present invention provides a simple, flexible method of obtaining a high yield of soft granulated material in optimum condition for subsequent charging to a pressing die or other shaping means. While specific materials and apparatus have been described for purposes of illustration, it will be appreciated that the invention is not so limited and may be carried out on finely divided materials generally and with any apparatus adaptable to the principles involved.

What is claimed is:

1. The method of granulating a finely divided ceramic powder which comprises mixing said powder with about the amount of water desired in the granulated product and enough more of an organic liquid that is more volatile than water and inert to said ceramic powder to form a plastic mass of the desired consistency, subdividing the plastic mass into coarse particles, introducing these particles over at least one screen of selected mesh size, causing the material to pass downwardly through said screen while simultaneously exposing the material to a current of air whereby the more volatile organic liquid is substantially removed and granulation of the material is effected.

2. A method as set forth in claim 1 in which the finely divided ceramic powder is uranium dioxide.

3. A method as set forth in claim 1 in which the finely divided ceramic powder is alumina.

4. A method as set forth in claim 1 in which the volatile organic liquid that is used is acetone.

5. A method as recited in claim 1 in which the coarse particles are introduced over and passed downwardly through a series of graduated screens of decreasing mesh size while being exposed to a counter-current of air.

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