

1

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PRODUCTION OF COMPRESSED PELLETS OR TABLETS FROM FINELY DIVIDED SOLID MATERIALS

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The present invention relates to the production of compressed pellets or tablets from finely divided solid materials.

The usual method of producing compressed pellets or tablets from finely divided materials is to feed the material into dies and compress it between punches, one of which, usually the lower one, is adapted to eject the compressed pellet and to return to its position for receiving the next charge of material.

Difficulty however, may arise due to a particular material inducing, during compression, excessive friction at the inner surfaces of the dies and thereby causing a large proportion of the applied pressure to be consumed without forming a satisfactory pellet.

Likewise difficulty often arises in ejecting the compressed pellets from the dies owing to the friction and/or adhesion between them and the inner surfaces of the dies, with the result that imperfect or fractured pellets are produced.

In attempts to overcome these difficulties a practice is to mix a lubricant with the material to be pelleted. Lubricants which have been used include finely divided long chain aliphatic hydrocarbons, alcohols, acids, esters and salts, also graphite and talc. Other substances having the characteristics of lubricants, such as the disulphides of molybdenum and tungsten, have been used where their presence can be tolerated.

This method, however, has obvious disadvantages as in many instances it may be very undesirable for the finished pellets to have added substances within them quite apart from the fact that the admixture of such substances to the material to be pelleted will, in general, adversely affect the mechanical strength of the finished pellets.

It has now been found that these disadvantages may be substantially overcome by the method of the present invention.

According to the present invention there is provided a process for the production of compressed pellets by feeding finely divided solid material into dies and compressing it between punches which comprises the successive steps of producing from each die at least one pellet of a material comprising a lubricant and then producing from each die at least one pellet of the desired material.

The number of pellets of the material comprising a lubricant to be produced from each die before producing pellets of the desired material will depend on the behaviour of the respective materials during pelleting, the amount and effectiveness of the lubricant in the first-mentioned material and also on the number of pellets of the desired material it is wished to produce before again producing pellets of the material comprising the lubricant.

It will be understood that preliminary trials will be necessary to determine the requisite data for any partic-

2

ular material or mixtures of materials with a desired lubricant.

It has been found that in pelleting some materials satisfactory results may be obtained when employing as little as ½% by weight of lubricant in the first-mentioned material, particularly if it is pelleted alternately (that is one for one) with the desired material.

A particular advantage of the process of the present invention is that less energy is consumed in overcoming the frictional resistances between the inner surfaces of the dies and the desired material and in consequence satisfactory pellets may be obtained with pressures much less than those required by the known method of mixing a lubricant with the desired material. It also follows that pellets of greater strength may be produced at pressures higher than those permissible with the known method, or the volume of desired material pelleted per unit time by a given machine may be substantially increased as larger pellets may be produced by the total pressure than can be exerted by the machine. For example it has been found that a machine, whose capacity is limited to ⅛" diameter × ⅛" long pellets when fed continuously with material containing a small percentage of graphite as lubricant, will produce satisfactory ⅜" diameter × ⅜" long pellets when each die is fed alternately with the same material containing graphite as lubricant and with similar material without graphite. Although the number of the larger pellets of the material containing no admixture of graphite is only one half of the total produced in a given time, their total volume is in the ratio of 27:16 compared with the number of the smaller ⅛" diameter × ⅛" long pellets that could be produced in the same time.

When the material to be pelleted is abrasive and/or requires high pressures to produce satisfactory results, it has been found preferable to pellet the desired material alternately with that comprising a lubricant. For example in preparing catalyst pellets comprising tungstic acid, nominally each ⅜" diameter × ⅜" long from granulated tungstic acid of particle size of 1.4 mm., it has been found that satisfactory pellets are obtained when the desired material is pelleted alternately with similar material which has been granulated after the addition of about 15% of powdered graphite as lubricant, and that the pressure required is then much less than that required to produce similar pellets by the known method of mixing the graphite with the desired material.

In the process of the present invention the pellets of the material comprising the lubricant may be crushed and re-used many times, if desired after the addition of fresh material and/or lubricant.

The process of the present invention is further illustrated in the following Tables 1 and 2 of test results obtained from ⅜" diameter x ⅜" long pellets of three powdered materials which may be used as catalysts and which for convenience are designated "A," "B" and "C":

- A being tungstic acid substantially of the formula H_2WO_4
B being a mixture of ferric oxide, chromic oxide and alumina in the ratio by weight of $Fe_2O_3:Cr_2O_3:Al_2O_3$ of 90:8:2
C being a mixture of zinc oxide and cupric oxide in the molar ratio of $2ZnO:1CuO$.

The data given in Table 1 are for the purpose of comparison with those given in Table 2 and are for pellets produced by the continuous pelleting of the materials mentioned in the first column when admixed with the percentage by weight of the lubricant given in the second column of the table. The percentages of lubricant shown are those which have been found desirable in admixture with the given materials in order to limit to a reasonable value the proportion of the punch pressure which is

absorbed in friction while, at the same time, obtaining pellets of good strength. The fourth column of Table 1 gives the mean percentage of the punch pressure which was absorbed in friction and the fifth column gives the mean vertical crushing strength in pounds of the finished pellets.

In Table 2 the second column gives the percentage by weight of lubricant which was used with the powdered material in the first column to produce one pellet from each die of the machine before producing pellets from the material without added lubricant.

It will be observed from the first entry for each material in Table 2 that the lubricant material per se was used as the initial pellet for one set of pellets of the lubricant-free material. It is to be understood that after completing each set of pellets the dies and punches of the machine were cleaned before the next set was produced.

The columns of Table 2 which are numbered from 1 to 12 give, in each instance, the number of satisfactory pellets produced from the lubricant-free material, their mean vertical crushing strength in pounds, and the percentage of the punch pressure which was absorbed in friction.

Table 1

Material	Lubricant	Punch pressure, tons/sq. in.	Friction, percent	M.V.C.S. (lb.)
"A"	1% graphite	80	20	650
"B"	2% graphite	60	24	340
"C"	1% graphite	60	27	310

at the lower punch pressure used with as much as 3% by weight of graphite in the material for the initial pellet.

We claim:

1. A process for the production of compressed pellets by feeding finely divided solid material into dies and compressing it between punches which comprises the successive steps of producing from each die at least one pellet of a material comprising a lubricant and then producing from each die at least one pellet of the desired material.

2. A process as claimed in claim 1 in which the lubricant is graphite.

3. A process for the production of compressed catalyst pellets of tungstic acid which comprises the steps of feeding finely divided tungstic acid, substantially of the formula H_2WO_4 , in admixture with at least 1/2% by weight of graphite, into dies and compressing it between punches to produce at least one pellet from each die and thereafter feeding finely divided tungstic acid free from graphite into the dies and compressing it between punches to produce at least one pellet from each die.

4. A process for the production of compressed catalyst pellets of a mixture of ferric oxide, chromic oxide and alumina in the ratio by weight of 90:8:2 which comprises the steps of feeding the finely divided mixture, in admixture with at least 3% by weight of graphite, into dies and compressing it between punches to produce at least one pellet from each die and thereafter feeding similar finely divided mixture free from graphite into the dies and compressing it between punches to produce at least one pellet from each die.

Table 2

Material	Lubricant, graphite	Punch Pressure, tons/sq. in.	Mean vertical crushing strength in lb. and Friction as percent load for Pellet Nos.—											
			1	2	3	4	5	6	7	8	9	10	11	12
"A"	Percent	100	900	820	980	960	960							
			13	13	15	16	17							
			1,025	925	925	975	825	750	500					
			14	15	17	17	18	20	23					
			950	875	900	925	750	500						
			15	16	18	20	24	33						
			875	1,000	900	875	950	375						
			16	17	18	19	24	34						
			950	950	900	1,000	475							
			16	17	19	21	26							
			600	560	580	580	500	520	430	350				
			14	14	17	19	23	30	36	42				
"B"	100	60	530	600	550	510	520	550						
			13	15	16	17	20	24						
			560	500	480	430								
			20	26	34	45								
			430	340										
			35	46										
			450	580	480	510	580	520	540	320				
			19	18	22	21	24	25	28	34				
			600	580	420	580	540	530	550	580	460	530	560	540
			15	16	16	17	18	19	20	19	22	23	25	26
			560	600	520	580	460	380	380					
			16	19	21	22	24	26	32					
"C"	100	60	460	460	480	440	400							
			19	20	25	27	33							

From Table 2 it is clear that the percentage of the punch pressure absorbed in friction was generally less and that the mean vertical crushing strength of the pellets was higher for those prepared in accordance with the invention than for those produced when using the known procedure of admixing lubricant with the material as shown in Table 1.

It is also clear from Table 2 that if the percentage of the punch pressure absorbed in friction as given in Table 1 is taken as the basis for satisfactory operation according to the invention, three or four pellets of material A may be produced from each die after producing an initial pellet of the material with as little as 1/2% by weight of graphite mixed therewith, but that with material C at least 1% by weight of graphite is required for similar results at the lower punch pressure used and that with material B only one or two pellets can be produced

5. A process for the production of compressed catalyst pellets of a mixture of zinc oxide and cupric oxide in the molar ratio of 2:1 which comprises the steps of feeding the finely divided mixture, in admixture with at least 1% by weight of graphite, into dies, and compressing it between punches to produce at least one pellet from each die and thereafter feeding similar finely divided mixture free from graphite into the dies and compressing it between punches to produce at least one pellet from each die.

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