

(19) World Intellectual Property Organization
International Bureau



(43) International Publication Date
21 December 2000 (21.12.2000)

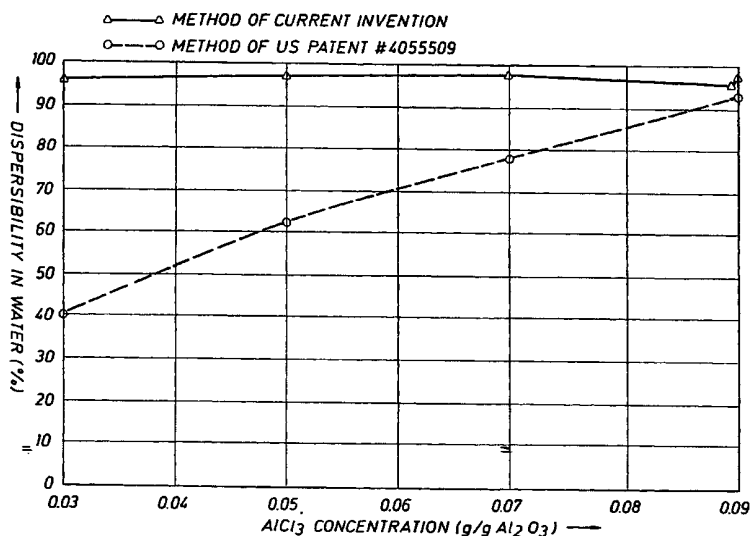
PCT

(10) International Publication Number
WO 00/76643 A1

- (51) International Patent Classification⁷: B01F 3/00, 3/12, C09K 3/00
- (21) International Application Number: PCT/US00/15856
- (22) International Filing Date: 9 June 2000 (09.06.2000)
- (25) Filing Language: English
- (26) Publication Language: English
- (30) Priority Data: 09/329,498 10 June 1999 (10.06.1999) US
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- (81) Designated States (*national*): AE, AL, AM, AT, AU, AZ, BA, BB, BG, BR, BY, CA, CH, CN, CR, CU, CZ, DE, DK, DM, EE, ES, FI, GB, GD, GE, GH, GM, HR, HU, ID, IL, IN, IS, JP, KE, KG, KP, KR, KZ, LC, LK, LR, LS, LT, LU, LV, MA, MD, MG, MK, MN, MW, MX, MZ, NO, NZ, PL, PT, RO, RU, SD, SE, SG, SI, SK, SL, TJ, TM, TR, TT, TZ, UA, UG, UZ, VN, YU, ZA, ZW.
- (84) Designated States (*regional*): ARIPO patent (GH, GM, KE, LS, MW, MZ, SD, SL, SZ, TZ, UG, ZW), Eurasian patent (AM, AZ, BY, KG, KZ, MD, RU, TJ, TM), European patent (AT, BE, CH, CY, DE, DK, ES, FI, FR, GB, GR, IE, IT, LU, MC, NL, PT, SE), OAPI patent (BF, BJ, CF, CG, CI, CM, GA, GN, GW, ML, MR, NE, SN, TD, TG).
- Published:
— With international search report.

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- For two-letter codes and other abbreviations, refer to the "Guidance Notes on Codes and Abbreviations" appearing at the beginning of each regular issue of the PCT Gazette.

(54) Title: METHOD FOR PRODUCING WATER-DISPERSIBLE ALPHA-ALUMINA MONOHYDRATE



(57) Abstract: A method of enhancing the dispersibility of alpha-alumina monohydrate wherein there is formed a mixture of an alpha-alumina monohydrate, water, and a dispersibility enhancer comprising an effective amount of a monobasic acid, a salt selected from the group consisting of salts having the formula MX wherein M is a polyvalent metal cation constituent for which the solubility product K_{SP} of the corresponding metal hydroxide is less than 10^{-20} and wherein X is an anion constituent derived from a monovalent acid having an ionization constant K_A greater than 10^{-1} and certain phosphorus and antimony chlorides, followed by drying the mixture, without aging, to produce a dried, alpha-alumina monohydrate having advanced water dispersibility.



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METHOD FOR PRODUCING WATER-DISPERSIBLE ALPHA-ALUMINA MONOHYDRATE

Background of the Invention

Field of the Invention

5 The present invention relates to water-dispersible alumina and, more particularly, to a method for producing an alpha-alumina monohydrate having enhanced water dispersibility.

Description of the Prior Art

As is well known, alpha-alumina monohydrate has widespread commercial usage
10 in such applications as abrasives, frictionizing paper surfaces, fiberglass surfaces, and metal surfaces; as static and soil protection agents on wool, nylon, and acrylic carpets; and as dispersing agents in rug shampoos, etc. In such commercial usages, the alumina is typically utilized as a dispersion in an aqueous acidic solution. It has long been known that adding acid to an aqueous alumina slurry that is dried to produce alpha-alumina
15 monohydrate results in a product that is dispersible in water to a high degree. Indeed, in U.S. Patent No. 4,676,928, incorporated herein by reference for all purposes, there is disclosed a method for enhancing the dispersibility of alumina in an acid solution by aging an alumina slurry containing a monobasic acid at elevated temperatures, e.g., at a temperature above about 70°C for a period of time sufficient to convert the greater
20 portion of the alumina to a colloidal sol. The advantage of the process disclosed in U.S. Patent No. 4,676,928 is that it produces an alumina having enhanced water dispersibility but with a generally lower acid content, thus avoiding the problems of corrosiveness and handling inherent in alumina compositions with high acid contents. However, the method still produces an acidic product and, moreover, requires extended aging at
25 elevated temperatures.

U.S. Patent No. 4,055,509, incorporated herein by reference for all purposes, discloses a process for increasing or enhancing the water dispersibility of alpha-alumina monohydrate by dry mixing alpha-alumina monohydrate with an effective amount of a salt compound, such as, for example, a salt having the formula MX wherein M is a
30 polyvalent metal cation constituent for which the solubility product K_{sp} of the

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corresponding metal hydroxide is less than 10^{-20} and wherein X is an anion constituent derived from a monovalent acid having an ionization constant K_A greater than 10^{+1} .

Summary of the Invention

It is therefore an object of the present invention to provide a process for producing an alpha-alumina monohydrate with enhanced water dispersibility.

Another object of the present invention is to provide a process for producing a dispersible alpha-alumina monohydrate that does not require aging an acidified alpha-alumina slurry at elevated temperatures.

Still a further object of the present invention is to provide a process for providing alpha-alumina monohydrates that can be dispersed in water to form substantially neutral sols.

The above and other objects of the present invention will become apparent from the drawings, the description given herein, and the appended claims.

According to the process of the present invention, there is formed a mixture of an alpha-alumina monohydrate, water, and a dispersibility enhancer comprising an effective amount of a salt compound selected from the group consisting of monobasic acids, salts having the formula MX wherein M is a polyvalent metal cation constituent for which the solubility product K_{sp} of the corresponding metal hydroxide is less than 10^{-20} and wherein X is an anion constituent derived from a monovalent acid having an ionization constant K_A greater than 10^{+1} , phosphorus dichloride, phosphorus trichloride, phosphorus pentachloride, phosphorus tribromide, phosphorus pentabromide, antimony pentabromide, and mixtures thereof, followed by drying the mixture at an elevated temperature sufficient to remove free water and produce a dried alpha-alumina monohydrate having enhanced water dispersibility.

Brief Description of the Drawings

Fig. 1 is a graph showing a comparison of the water dispersibility of an alpha-alumina monohydrate produced by the process of the present invention with an alpha-alumina monohydrate produced by the process of U.S. Patent No. 4,055,509;

Fig. 2 is a graph comparing the water dispersibility of an alpha-alumina monohydrate sol containing aluminum nitrate with dried solids or powder produced from

the sol in accordance with the process of the present invention;

Fig. 3 is a graph illustrating the relationship between dispersibility, crystallite size, and aluminum nitrate concentration of alpha-alumina monohydrate having enhanced dispersibility prepared according to the process of the present invention; and

5 Fig. 4 illustrates the effect of aluminum nitrate concentration on dispersibility.

Description of the Preferred Embodiment

The aluminas that can be treated according to the process of the present invention are alpha-alumina monohydrates that are generally apparently dry products that comprise alpha-alumina monohydrate ($\text{Al}_2\text{O}_3 \cdot \text{H}_2\text{O}$) and in addition may contain free water and the
10 like. Such alpha-alumina monohydrates are commonly referred to as pseudo-boehmite or boehmite aluminas. The extent to which the alpha-alumina monohydrate has been dried is indicated by the Al_2O_3 content of the monohydrate. Typically, the alpha-alumina monohydrate commercially available contains less than about 86% by weight Al_2O_3 and, in most instances, contains less than about 81% by weight Al_2O_3 . More typically, the
15 Al_2O_3 content of commercially available alpha-alumina monohydrates ranges from about 68 to about 75% by weight.

From a dispersibility point of view, when the alpha-alumina monohydrate is dried such that the Al_2O_3 content approaches 86% by weight, the product is difficult to extrude, disperse, or otherwise use in any form other than that in which it exists prior to the
20 drying. It is well known that when all the free water has removed from the apparently dry alpha-alumina monohydrate dispersion, extrusion, dispersion, etc., become extremely difficult, and accordingly, most alpha-alumina monohydrate is marketed in a weight range of from 68 to about 75% by weight Al_2O_3 . However, even at concentrations as low as 50% by weight Al_2O_3 , the alpha-alumina monohydrate, which appears to be a dry
25 solid, is not readily dispersible in water. In this regard, dispersibility on the order of 25 to 55% by weight is common, and when the alpha-alumina monohydrate is dried in the range as set forth above--i.e., 68 to 75% by weight Al_2O_3 --water-only dispersibility on the order of 5 to 20% is not uncommon. As noted above, the dispersibility of the monohydrate alumina can be markedly enhanced by the use of acids.

30 The alpha-alumina monohydrates useful in the process of the present invention can be produced by techniques such as the water hydrolysis of aluminum alkoxides, the

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sodium aluminate process, and the alum process, all of which are well known to those skilled in the art. However, it has been found that aluminas obtained by the hydrolysis of aluminum alkoxides are particularly desirable for use in the process of the present invention. Processes for preparing and hydrolyzing aluminum alkoxides are disclosed in U.S. Patent Nos. 4,242,271 and 4,202,870, incorporated herein by reference for all purposes.

The dispersibility enhancers that have been found to be useful in the process of the present invention include monobasic acids, such as nitric acid, hydrochloric acid, and organic acids having from 1 to 2 carbon atoms, such as formic acid, acetic acid, and the like, as well as salt compounds selected from the group consisting of salts having the formula MX wherein M is a polyvalent metal cation constituent for which the solubility product K_{sp} of the corresponding metal hydroxide is less than 10^{-20} and wherein X is an anion constituent derived from a monovalent acid having an ionization constant K_A greater than 10^{+1} . Additionally, compounds such as phosphorus dichloride, phosphorus trichloride, phosphorus pentachloride, phosphorus tribromide, phosphorus pentabromide, and an antimony pentachloride have also been found to be useful. Particularly preferred of the foregoing are the monobasic acids, such as nitric acid and hydrochloric acid, nitric acid being especially preferred. Also preferred are salts of the formula MX wherein the cations consist of Sc^{+3} , Y^{+3} , Nd^{+3} , Sn^{+3} , Eu^{+3} , Gd^{+3} , Er^{+3} , Tm^{+3} , Yb^{+3} , Lu^{+3} , Ti^{+3} , Zr^{+4} , Th^{+3} , Cr^{+3} , U^{+4} , Mn^{+3} , Fe^{+3} , Co^{+3} , Ru^{+3} , Pd^{+2} , Pt^{+2} , Au^{+3} , Al^{+3} , Ga^{+3} , In^{+3} , Sn^{+2} , Sn^{+4} , Bi^{+3} and the anions are selected from the group consisting of Cl^- , NO_3^- , I^- , ClO_3^- , ClO_4^- , Br^- , MnO_4^- . Of the phosphorus halide salts listed above, the chloride salts are preferred.

Generally, the mixture of the alpha-alumina monohydrate/dispersibility enhancer/water will contain from about 5 to about 15% by weight alumina calculated as Al_2O_3 . The dispersibility enhancers are used in an effective amount, which is an amount necessary to impart the desired degree of dispersibility to the alumina. Accordingly, the amount of the dispersibility enhancers in the mixture can vary widely but will generally range from about 0.5 to about 10% by weight, based on the weight of the alpha-alumina monohydrate calculated as Al_2O_3 .

According to the process of the present invention, an alpha-alumina monohydrate as described above, one or more of the dispersibility enhancers as described above, and water are brought together to form a mixture, generally the dispersibility enhancers being

soluble in the water such that there remains an aqueous solution of the dispersibility enhancer and solid alumina particles, which can be in colloidal form. In the prior art processes described in U.S. Patent No. 4,676,928, and when a monobasic acid was employed, it was necessary for the slurry of alumina and the acid to be aged at a temperature of at least about 70°C, more generally from about 70 to about 100°C. Generally speaking, the aging was conducted for a period of time sufficient to convert the alumina in the slurry to a colloidal sol, the time varying with pH and temperature. However, as seen by the examples in U.S. Patent No. 4,676,928, aging times of several hours up to several months were necessary. Thus, unlike the process disclosed in U.S. Patent No. 4,676,928, no aging of the alpha-alumina monohydrate/dispersibility enhancer/water mixture is required. Rather, the mixture is simply dried, e.g., by spray drying or in an oven, at a temperature sufficient to remove substantially all free water and produce a dried solid or powder, the form of the dried material depending upon the type of drying employed. Generally, the drying temperature will range from 60 to 110°C, although higher temperatures can be used provided bound water is not removed--i.e., the monohydrate is not calcined.

The method of the present invention provides several distinct advantages over the prior art techniques discussed above with respect to U.S. Patent Nos. 4,055,509 and 4,676,928 for enhancing water dispersibility. For one, and as noted above, it is unnecessary to employ long aging times at elevated temperatures. With respect to the method disclosed in U.S. Patent No. 4,055,509, the present process is a distinct improvement in that the alpha-alumina monohydrate produced has much greater dispersibility and is achieved at lower dispersibility enhancer concentrations.

The method of the present invention can be applied to alumina slurries or alumina powder that is slurried in water. Several distinct benefits flow from the process of the present invention. Since heat treating is not necessary, reaction times can be reduced by growing crystals in the absence of acid and then adding the dispersibility enhancer (salt) once the appropriate crystallite size is reached. Additionally, one can produce a neutral alpha-alumina monohydrate sol since no acid is added when the dispersibility enhancer is one of the salts mentioned above. Using the present invention, one can also disperse small crystallite size alumina without high acid addition. The process of the present invention is also readily adaptable to current continuous processes for producing alpha-

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alumina monohydrates from the hydrolysis of aluminum alkoxides since the dispersibility enhancers can be added into the alumina slurry recovered from the hydrolysis reaction prior to being sent to a spray drier. Lastly, it is believed that the present invention allows the production of doped aluminas exhibiting catalytic properties. In this regard, zirconium and other metal ions that exhibit catalytic activity in a number of reactions can be incorporated into the alumina by adding the zirconium-containing compound that meets that dispersibility enhancing criteria set out above into the alumina slurry recovered from the hydrolysis reaction such that the mixture of the alumina slurry and the zirconium compound, when spray dried, constitutes a catalytically active species. To more fully illustrate the present invention, the following non-limiting examples are presented.

Example 1

A mixture of 25 grams of CATAPAL® B alpha-alumina monohydrate powder marketed by CONDEA Vista Company was mixed with 175.5 grams of water and varying amounts of AlCl_3 . The mixtures were dried by pan-drying at 60°C for several hours until a solid containing no free water was obtained. Following the method of U.S. Patent No. 4,676,928, dry mixtures of the CATAPAL® B alumina and various amounts of aluminum chloride were also formed by blending the two ingredients together to form a generally homogeneous, dry mixture. The dispersibility in water of the aluminas prepared according to the present invention and the aluminas prepared according the method of U.S. Patent No. 4,055,509 was then compared. The results are shown in Fig. 1. As can be seen from Fig. 1, using the process of the present invention, the alumina has a dispersibility in water of greater than 95% with a range of 0.03 to 0.09 grams of AlCl_3 per g of Al_2O_3 , whereas in the case of the alumina prepared by the method of U.S. Patent No. 4,055,509, dispersibility ranged from 40% by weight when the AlCl_3 content was 0.03 grams per gram of Al_2O_3 to greater than 90% when the concentration of AlCl_3 was 0.09 grams per gram of Al_2O_3 . This example clearly demonstrates that, compared to the method of U.S. Patent No. 4,055,509, a much smaller amount of salt can be used to obtain a highly dispersible alumina.

Example 2

Sols comprised of water, aluminum nitrate, and CATAPAL® D, an alpha-alumina monohydrate marketed by CONDEA Vista Company, were prepared. The aluminum nitrate concentrations ranged from 0.06 to 0.10 g $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}/\text{g Al}_2\text{O}_3$.

5 In two cases, the sols were dried either by pan-drying at 60°C for several hours or spray drying at an inlet temperature of 340°C and an outlet temperature of 110°C with a residence time of a few seconds. In a third case, the dispersibility of the CATAPAL® D alumina in the sol (undried) was determined. The results are shown in Fig. 2. As can be seen from Fig. 2, in the undried sol of CATAPAL® D alumina and aluminum nitrate,

10 the dispersibility reaches a maximum of 77% at a concentration of approximately 0.17 grams of $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}/\text{g Al}_2\text{O}_3$, whereas in the case where the sol was dried, either by oven drying or spray drying, dispersibility increased dramatically to as much as 98.2%. More importantly, throughout the entire range of aluminum nitrate addition, dispersibility was greater than 95% in the case of the dried materials, whereas in the case

15 of the aluminum sol itself, dispersibility ranged from a low of 54.7% to a high of 77.0%. It is particularly to be noted that at lower concentrations of the aluminum nitrate, the difference in dispersibility was particularly dramatic, ranging from approximately 57% at 0.06 g $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}/\text{g Al}_2\text{O}_3$ to greater than 97% at the same aluminum nitrate/alumina ratio.

20 Example 3

This example demonstrates the dependence of dispersibility on the crystallite size and the dispersibility enhancer concentration. Mixtures of alpha-alumina monohydrates of various crystallite sizes were mixed with given amounts of aluminum nitrate and pan-dried at 60°C to remove free water. The resulting powder was then mixed with water to

25 determine the dispersibility. The results are shown in Fig. 3. As can be seen from Fig. 3, as the crystallite size of the alumina increases, the dispersibility increases. Like results are seen with respect to the aluminum nitrate concentration; i.e., as aluminum nitrate concentration increase, dispersibility increases.

Example 4

This example demonstrates the effect of the concentration of dispersibility enhancer on dispersibility. Three sets of alpha-alumina monohydrate samples with various crystallite sizes were employed. One was an experimental sample having a
5 crystallite size, 020 plane, of 60 Å, one was a CATAPAL® D alumina having a crystallite size, 020 plane, of 41 Å, and one was a CATAPAL® A alumina having a crystallite size, 020 plane, of 28 Å. The slurries of the alumina described above were mixed with varying amounts of aluminum nitrate and then pan-dried at 60°C. The results are shown in Fig. 4. As can be seen from Fig. 4, at low concentrations of
10 aluminum nitrate, the greater the crystallite size, the greater the dispersibility. Further, as the concentration of aluminum nitrate increases, the dispersibility increases. As can also be seen from Fig. 4, there appears to be a relatively sharp transition region where the dispersibility rises significantly for small changes in aluminum nitrate concentration. This concentration region is dependent on crystallite size. For example, the sharp
15 increase in dispersibility occurs over an aluminum nitrate concentration range of 0.03 to 0.08 g $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}/\text{g Al}_2\text{O}_3$ for an alumina with a crystallite size of 28 Å (020plane). However, the transition region for an alumina with a crystallite size of 41 Å occurs over a concentration range of 0.01 to 0.03 g $\text{Al}(\text{NO}_3)_3 \cdot 9\text{H}_2\text{O}/\text{g Al}_2\text{O}_3$.

Example 5

20 This example demonstrates dispersibility results achieved by the process of the present invention using a variety of dispersibility enhancers. Aqueous sols of CATAPAL® B and various dispersibility enhancers were prepared. The dispersibility of the alumina in the sol was measured, as well as the dispersibility of the powders obtained by pan-drying the sols at 60°C. The results are shown in Table 1 below.

Table: Effect of Various Other Additives on Dispersibility of CATAPAL® B

Additive	Concentration 1 (g additive/g Al ₂ O ₃)	Sol Dispersibility (%)	Powder Dispersibility (%)	Concentration 2 (g additive/g Al ₂ O ₃)	Sol Dispersibility (%)	Powder Dispersibility (%)
HNO ₃	0.050	51.4	97.0	0.100	98.1	98.2
Fe(NO ₃) ₂ •9H ₂ O	0.051	<10	33.8	0.102	18.1	97.0
ZrO(NO ₃) ₃ (in HNO ₃)	0.037	12.3	53.6	0.076	51.2	92.8
La ₂ (NO ₃) ₃	0.037	10.1	22.6	0.073	<10	23.0
Ce ₂ (NO ₃) ₃	0.037	12.1	16.4	0.075	11.7	21.4
NaNO ₃	0.050	<10	16.7	0.101	<10	13.7
FeCl ₃ •6H ₂ O	0.050	<10	49.3	0.100	37.8	98.3
HCl	0.025	36.2	95.1	0.050	78.7	98.2
NaCl	0.050	<10	<10	0.100	<10	<10
Fe ₂ (SO ₄) ₃ •xH ₂ O	0.050	<10	<10	0.100	<10	<10
Al ₂ (SO ₄) ₃ •xH ₂ O	0.050	<10	<10	0.100	<10	<10
Al(C ₂ H ₃ O ₂) ₃	0.050	<10	25.3	0.100	<10	27.8

As can be seen from the data, powders obtained by the process of the present invention wherein the sol containing the dispersibility enhancer is dried show markedly greater water dispersibility as compared with the sols themselves. As can further be seen, combinations of ions such as Fe^{+3} and NO_3^{-1} will disperse alumina, but combinations of ions such as Na^{+1} and NO_3^{-1} will not. It is also apparent that multivalent anions such as SO_4^{-2} are not suitable for dispersing alumina. Table 1 also demonstrates that dramatic increases in dispersibility occur when using acids as dispersibility enhancers. For example, at a concentration of 0.025 g HCl/g Al_2O_3 , the alumina dispersibility increases from 36% in the sol to 95 % of the powder after drying the sol and re-dispersing the powder.

As the above examples clearly demonstrate, the process of the present invention provides a markedly improved method for increasing the dispersibility of alpha-alumina monohydrate. The method does not require long-term aging or aging at elevated temperatures and therefore is much more economical than processes such as disclosed in U.S. Patent No. 4,676,928. Additionally, markedly enhanced dispersibility can be achieved without the use of acids such that neutral, dispersible aluminas can be prepared. Furthermore, the present invention is a marked improvement over the method disclosed in U.S. Patent No. 4,055,509 inasmuch as lower concentrations of dispersibility enhancers, e.g., salts, are required. Additionally, as compared to the method of U.S. Patent No. 4,055,509, which requires dry blending or mixing, the process of the present invention, which uses an aqueous dispersion, ensures a more homogeneous product.

The foregoing description and examples illustrate selected embodiments of the present invention. In light thereof, variations and modifications will be suggested to one skilled in the art, all of which are in the spirit and purview of this invention.

What is claimed is:

1. A method of increasing the dispersibility of alpha-alumina monohydrate comprising:
 - forming a mixture of alpha-alumina monohydrate, water, and a dispersibility enhancer selected from the group consisting of monobasic acids, salts having the formula MX, wherein M is a polyvalent metal cation constituent for which the solubility product K_{sp} of the corresponding metal hydroxide is less than 10^{-20} and wherein X is an anion constituent derived from a monovalent acid having an ionization constant K_A greater than 10^{-1} , phosphorus dichloride, phosphorus trichloride, phosphorus pentachloride, phosphorus tribromide, phosphorus pentabromide, and mixtures thereof; and
 - drying said mixture at a temperature sufficient to substantially remove free water and produce a dried powder of alpha-alumina monohydrate having enhanced water dispersibility.
2. The process of Claim 1 wherein M is a polyvalent metal cation constituent selected from the group consisting of Sc^{+3} , Y^{+3} , Nd^{+3} , Sn^{+3} , Eu^{+3} , Gd^{+3} , Er^{+3} , Tm^{+3} , Yb^{+3} , Lu^{+3} , Ti^{+3} , Zr^{+4} , Th^{+3} , Cr^{+3} , U^{+4} , Mn^{+3} , Fe^{+3} , Co^{+3} , Ru^{+3} , Pd^{+2} , Pt^{+2} , Au^{+3} , Al^{+3} , Ga^{+3} , In^{+3} , Sn^{+2} , Sn^{+4} , Bi^{+3} , and wherein x is an anion constituent derived from a monovalent acid and is selected from the group consisting of Cl^- , NO_3^- , I^- , ClO_3^- , ClO_4^- , Br^- , MnO_4^- .
3. The process of Claim 1 wherein said monobasic acid is selected from the group consisting of nitric acid, hydrochloric acid, and organic acids having from 1 to 2 carbon atoms.
4. The process of Claim 1 wherein M is selected from the group consisting of Sn^{+3} , Ti^{+3} , Cr^{+3} , Mn^{+3} , Fe^{+3} , Co^{+3} , Pd^{+2} , Pt^{+2} , Au^{+3} , Al^{+3} , Sn^{+2} , Sn^{+4} , and Bi^{+3} .
5. The method of Claim 4 wherein X is selected from the group consisting of Cl^- , NO_3^- , I^- , and Br^- .

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6. The method of Claim 1 wherein said dispersibility enhancer is selected from the group consisting of phosphorus dichloride, phosphorus trichloride, phosphorus pentachloride, and antimony pentachloride.

7. The process of Claim 1 wherein said alumina is present in said mixture
5 in an amount of from 5 to 15% by weight calculated as Al_2O_3 and said dispersibility enhancer is present in said mixture in an amount of from 0.5 to about 10% by weight, based on the weight of the alpha-alumina monohydrate calculated as Al_2O_3 .

8. The process of Claim 1 wherein said alpha-alumina monohydrate contains up to 81% by weight Al_2O_3 .

9. The process of Claim 1 wherein said alpha-alumina monohydrate contains
10 up to 78% by weight Al_2O_3 .

10. The process of Claim 1 wherein said alpha-alumina monohydrate contains from 68 to 78% by weight Al_2O_3 .

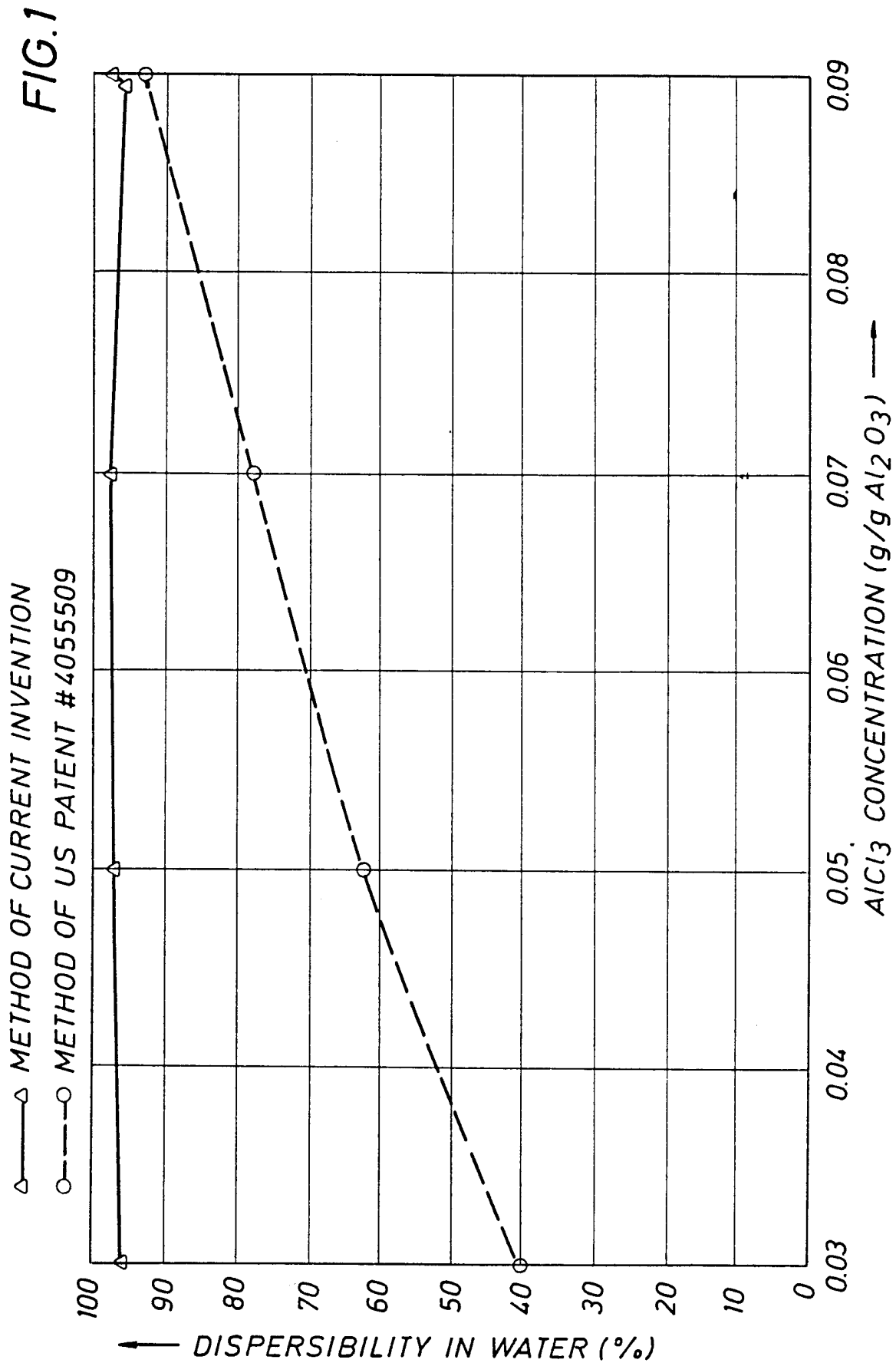


FIG. 2

○ SOL. DISPERSIBILITY BEFORE DRYING

□ OVEN-DRIED SOLIDS

△ SPRAY-DRIED POWDER

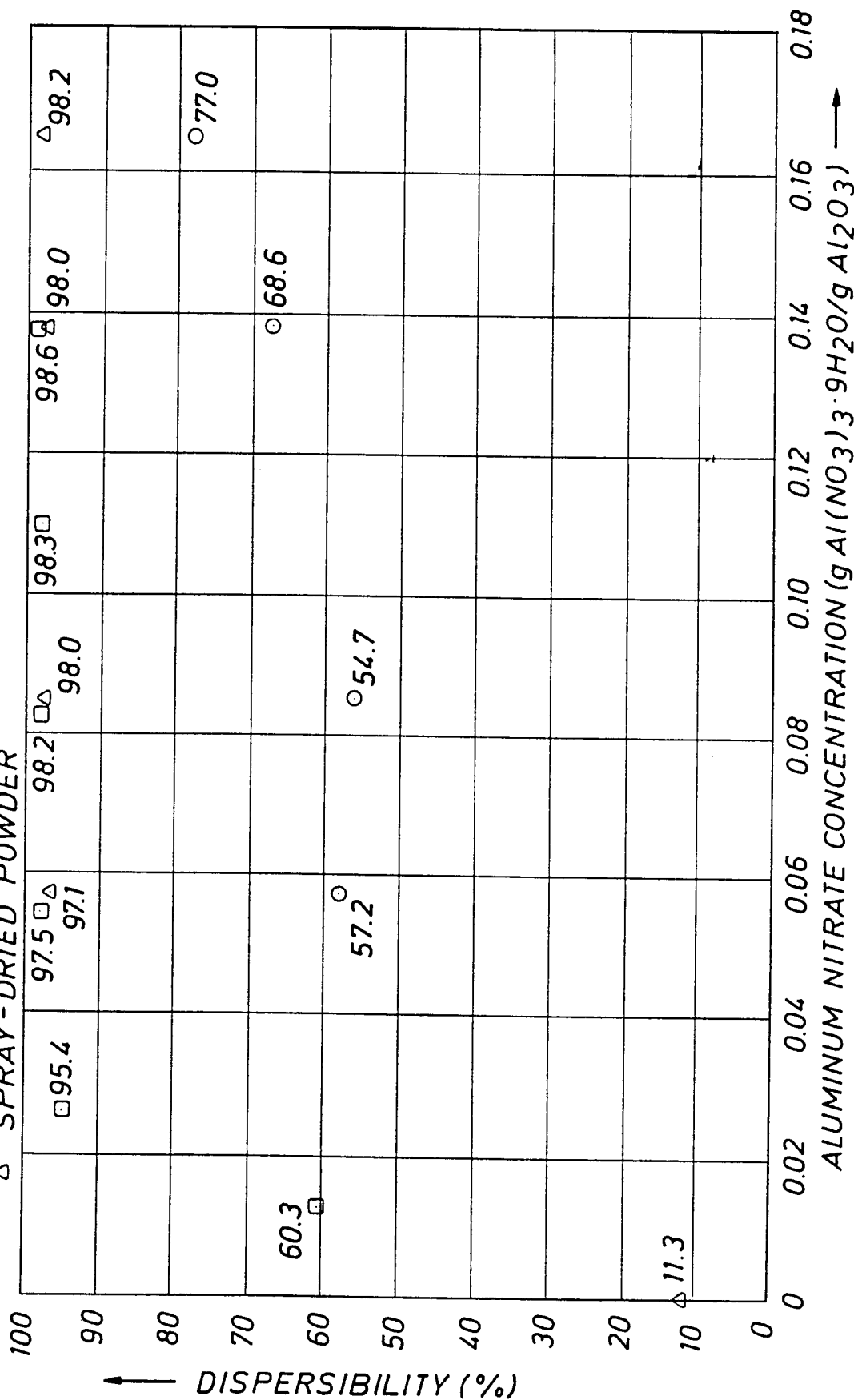
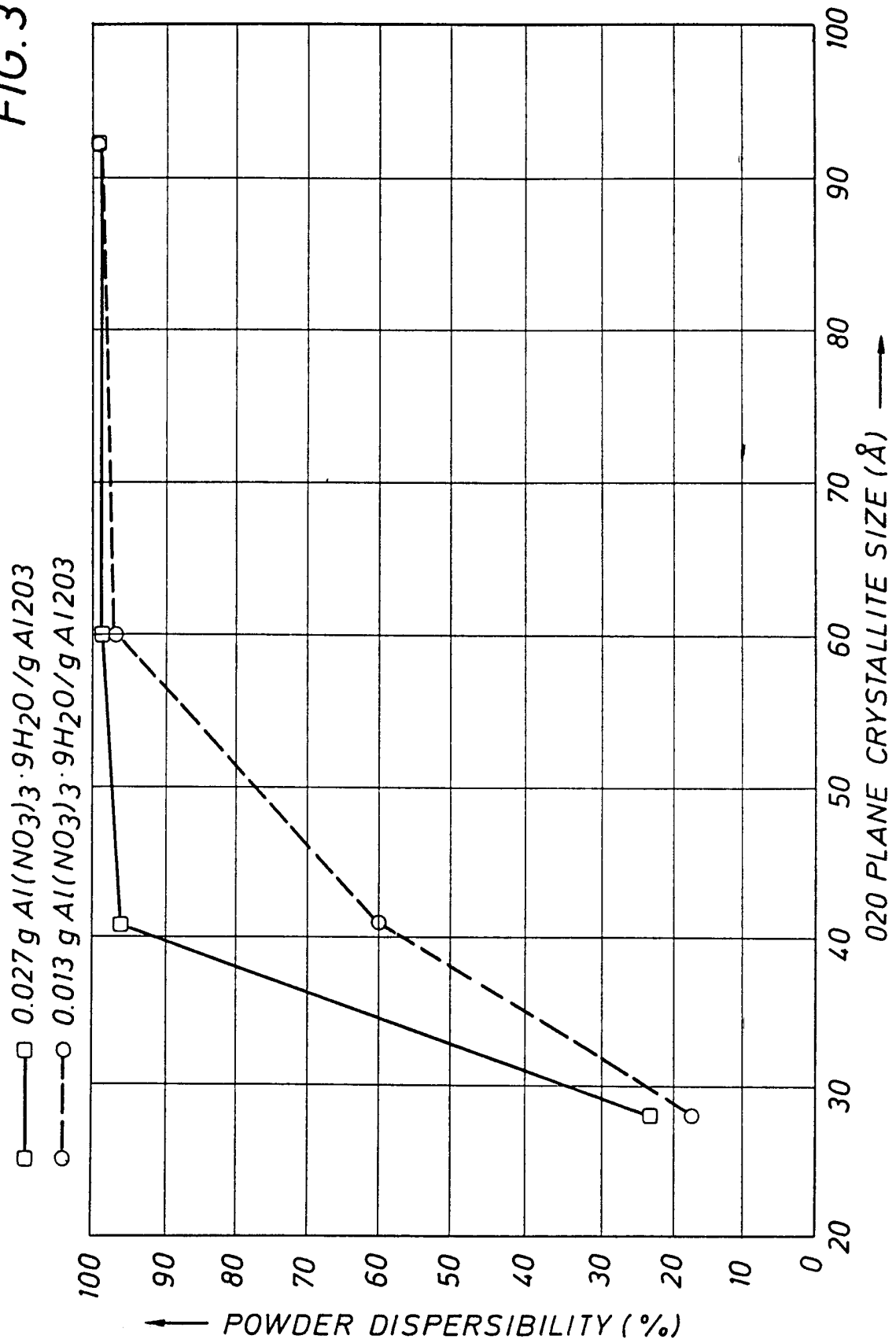
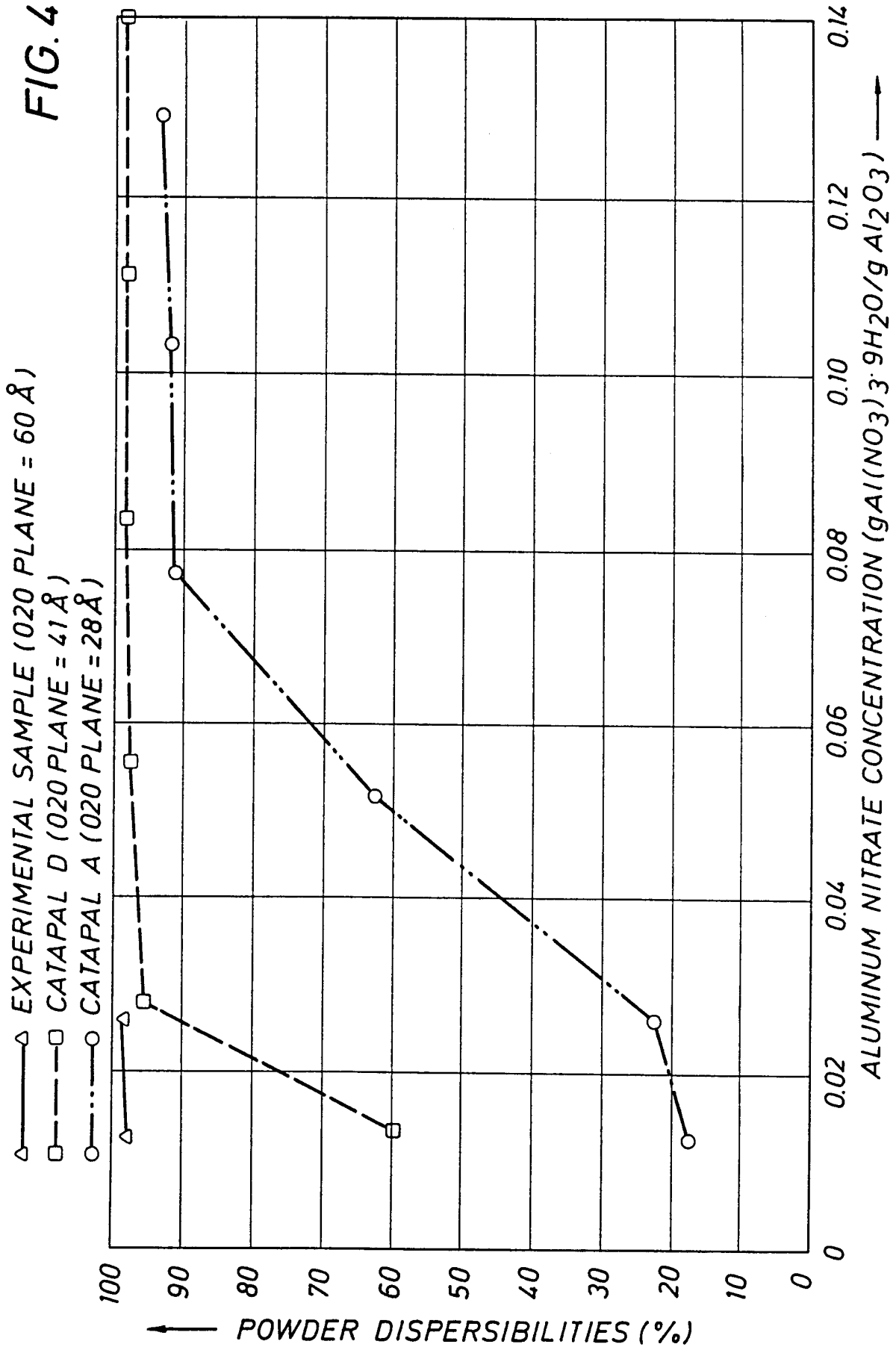


FIG. 3





INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/15856

A. CLASSIFICATION OF SUBJECT MATTER

IPC(7) : B01F 3/00, 3/12; C09K 3/00
US CL : 252/363.5

According to International Patent Classification (IPC) or to both national classification and IPC

B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 252/363.5

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

WEST 2.0


C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X	US 3,975,509 A (ROYER et al) 17 August 1976, col. 2, lines 27-33, col. 3, lines 5-14, and examples.	1, 3, 7-10
X	US 4,666,614 A (BLOCK) 19 May 1987, see abstract and examples.	1, 3
Y	US 4,055,509 A (WEIMER) 25 October 1977, col. 2, lines 6-18.	2, 4-6
Y	US 5,178,849 A (BAUER) 12 January 1993, col. 6, line 65 - col. 8, line 7.	1, 3
A	US 2,260,871 A (SAWYER) 28 October 1941, see entire document.	
A	US 4,244,835 A (BLOCK) 13 January 1981, see entire document.	

Further documents are listed in the continuation of Box C. See patent family annex.

<p>* Special categories of cited documents:</p> <p>"A" document defining the general state of the art which is not considered to be of particular relevance</p> <p>"E" earlier document published on or after the international filing date</p> <p>"L" document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)</p> <p>"O" document referring to an oral disclosure, use, exhibition or other means</p> <p>"P" document published prior to the international filing date but later than the priority date claimed</p>	<p>"T" later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention</p> <p>"X" document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone</p> <p>"Y" document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art</p> <p>"&" document member of the same patent family</p>
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Date of the actual completion of the international search 16 JULY 2000	Date of mailing of the international search report 17 AUG 2000
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INTERNATIONAL SEARCH REPORT

International application No.
PCT/US00/15856

C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 5,837,634 A (MCLAUGHLIN et al) 17 November 1998, see entire document.	