

EFFECT OF THE REMOVAL OF REACTIVE SURFACE MATERIAL ON THE SOLUBILITY OF SYNTHETIC GIBBSITES¹

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Abstract—The solubility of four different gibbsite preparations was measured, including two commercial hydrated aluminas produced by Alcoa, a Fisher ACS-grade $\text{Al}(\text{OH})_3$, and a laboratory preparation. The Alcoa samples and the laboratory-prepared sample had been studied previously by other investigators but without using a long-term acid treatment. Scanning electron microscopy showed globular surface material that was removed by a 14-day, 0.1 M HCl treatment. Solubility was determined at pH 4 from both over- and undersaturation with continuous agitation for 228 days. The acid treatment correlated with a decrease in $\log^*K_{so} [^*K_{so} = (\text{Al}^{3+})/(\text{H}^+)^3]$ of about 0.5 units. The mean solubility of the three acid-treated commercial gibbsite samples was $\log^*K_{so} = 7.55 \pm 0.055$ ($^*K_{so} = 3.5 \times 10^7$). The solubility of the acid-treated laboratory preparation was $\log^*K_{so} = 7.86$ ($^*K_{so} = 7.2 \times 10^7$). The greater solubility of the laboratory-prepared gibbsite is attributed to a greater concentration of structural defects in the crystals.

Key Words—Acid solubility, Gibbsite, Scanning electron microscopy, Solution, Structural defects.

INTRODUCTION

The solubility of gibbsite in acid solutions has been investigated repeatedly. Long-term experiments with highly crystalline gibbsite yielded \log^*K_{so} [$^*K_{so} = (\text{Al}^{3+})/(\text{H}^+)^3$] values at 298°K of 8.0 (Kittrick, 1966; Singh, 1972) to 8.5 (Frink and Peech, 1962). Kittrick (1966) argued that the greater solubility measured by Frink and Peech (1962) was due to the lower crystallinity of the gibbsite used. Our examination of both the gibbsite samples used by Kittrick (1966) and the one used by Frink and Peech (1962) with scanning electron microscopy (SEM), showed that all samples have surface coatings of finely divided, globular material, suggesting the possibility that they contained poorly crystalline $\text{Al}(\text{OH})_3$. The purpose of the present study was to investigate the effect of removing the surface material on the solubility of several gibbsite samples, including those used by Frink and Peech (1962) and Kittrick (1966). Preliminary investigation showed that a short-term, acid-wash similar to that used by Frink and Peech (1962) and May *et al.* (1979) removes very little Al. Therefore, a 14-day equilibration in 0.1 M HCl was used as a pretreatment before the solubility determinations.

MATERIALS AND METHODS

Gibbsite samples

Five gibbsite samples, C-33, C-730, FF, FC, and L were used. The specific surface area and sodium con-

tent of the samples are shown in Table 1. The specific surface area was determined by BET- N_2 adsorption with a Quantasorb Surface Area Analyzer (Quantachrome, Inc.). The sodium content of the untreated samples was determined using flame photometry after dissolution in HF. The method was similar to the method of Jackson (1969) for total analysis of silicate clays.

Samples C-33 and C-730 are commercial hydrated alumina produced by Alcoa Corporation. These samples had been used by Kittrick (1966) for his solubility determinations. Differential thermal analysis (DTA) and X-ray powder diffraction (XRD) showed that both samples are well-crystalline gibbsite (Kittrick, 1966). The XRD pattern agrees well with the Joint Committee on Powder Diffraction Standards (JCPDS) file 7-324 for gibbsite, and the DTA pattern agrees well with the data for gibbsite in the *Data Handbook for Clay Materials and Other Non-Metallic Minerals* (Van Olphen and Fripiat, 1979). The manufacturer reported Fe_2O_3 contents of 0.003 and 0.02% and SiO_2 contents of 0.02 and 0.05% for samples C-33 and C-730, respectively. The average particle diameter of sample C-730 reported by Alcoa is 0.05 μm . The reported screen analysis of sample C-33 is 92–100% to pass 100 mesh, with 60–80% to pass 325 mesh. Samples FF and FC are different particle-size fractions separated from the same batch of Fisher ACS $\text{Al}(\text{OH})_3$. The two fractions were separated by sedimentation in water to yield >20- μm and 0.2–20- μm samples of FF and FC, respectively. The manufacturer reported a trace of SiO_2 , <0.05% SO_4 , <0.005% Fe_2O_3 , and <0.005% other heavy metals. Our XRD pattern agreed well with the JCPDS file 7-324.

Sample L was a laboratory preparation by W. L.

¹ Scientific Journal Series, Paper No. 11,682.

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Table 1. Surface area and percent Na of gibbsite samples.

Samples	Source	Surface area ¹ m ² /g	Na content ² %
C-730	Alcoa	7.51	0.42
C-33	Alcoa	0.385	0.17
FF	Fisher Al(OH) ₃	1.82	0.18
FC	Fisher Al(OH) ₃	0.411	0.28
L	W. L. Lindsay	N.D.	0.26

¹ Determined at the end of the solubility experiment.

² Determined on the untreated samples.

Lindsay.³ This gibbsite was used by Frink and Peech (1962) in their determination of gibbsite solubility. Petrographic examination by Lindsay showed only well-crystalline gibbsite (Frink and Peech, 1962). The sample size was insufficient for further XRD and chemical analyses. Frink and Peech (1962) reported a particle size of 2–6 μm. Lindsay's original notation, however, indicates a particle size of 2–60 μm. This is consistent with the scanning electron microscopic observations.

Scanning electron microscopy (SEM)

Aqueous suspensions of gibbsite were deposited on carbon-coated aluminum stubs. The samples were coated with gold-palladium prior to examination with a Philips 500 scanning electron microscope.

Determination of solubility

Ten grams of samples C-33 and FF and 2 g of samples C-730 and LL were pretreated by shaking them in 250-ml polyethylene bottles with 100 ml and 200 ml, respectively, of 0.1 M HCl for 14 days. The gibbsite was filtered and washed with 100-ml volumes of H₂O until the pH ≥ 4.2. The filtrate was analyzed for Al and Na. The solubility of all samples, except L, was determined from the initial conditions of both undersaturation and oversaturation with respect to the solubility of gibbsite as determined by Kittrick (1966). Because of the small sample size, the solubility of sample L was determined from oversaturation only. Undersaturated conditions were established by adding HCl sufficient to make the solutions 2.1 × 10⁻⁴ M with respect to H⁺ (pH = 3.7). Oversaturated conditions were established by adding AlCl₃ sufficient to make the solutions 1.5 × 10⁻⁴ M with respect to Al. The quantities of HCl and AlCl₃ used were chosen to yield a calculated equilibrium pH of 4.0. The bottles were agitated continuously on a slow reciprocating bed shaker. After 3, 19, and 229 days, solution samples were taken after high-speed centrifugation for determination of pH and Al. All treatments were in duplicate. The laboratory temperature was 295 ± 1°K at each sampling. The temperature at the

Table 2. Percent of Al and Na in gibbsite samples removed by 14-day 0.1 M HCl treatment.

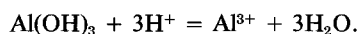
Sample	Al removed (%)	Na removed (%)
C-730	10.5	6.6
C-33	0.61	0.67
FF	3.9	27
L	3.9	7.3

time of the analysis of the solution samples from the 228-day treatment was 295.0 ± 0.50°K.

Three 10-g samples of untreated sample FC were suspended in 200 ml of 0.01 M KNO₃ in 250-ml polyethylene bottles. Two oversaturated suspensions were prepared by making one solution 2 × 10⁻³ M and the other 1 × 10⁻⁴ M with respect to AlCl₃. The third suspension was made undersaturated by adding 2.5 × 10⁻⁴ M HCl. The suspensions were agitated in an end-over-end shaker and analyzed after 1, 7, and 28 months. The temperature at the time of the 28-month analysis was 295 ± 1°K.

Aluminum was determined spectrophotometrically by the 8-hydroxyquinoline method of Bloom *et al.* (1978). The pH was determined to ±0.02 units using a Corning Model 12 expanded scale pH meter with a saturated calomel reference electrode equipped with a flowing liquid junction. The electrodes were calibrated using pH 4.01, 6.86, and 7.41 buffers.

Solution Al³⁺ activity was calculated from the analytical concentrations using the extended Debye-Huckel equation and the formation constant for AlOH²⁺ (Frink and Peech, 1963). The solubilities were expressed in units of log*K_{so}, where *K_{so} is the equilibrium constant for the reaction:



RESULTS

Scanning electron microscopy

Examination of the samples with SEM showed that the reported particle size data are not reflective of the size of single, well-formed crystals (Figures 1 and 2). Twinning is common in sample C-730, whereas agglomeration resulting from cementation and intergrowth is common in the other samples.

Untreated samples contained abundant globular surface material (Figure 1). Much of this surface material was removed by the 14-day equilibration in 0.1 M HCl (Figure 2). Analysis of the acid-wash solution (Table 2) showed that, with the exception of sample FF, the percentage of Na removed by the wash solution was similar to the percentage of Al removed, suggesting that the surface material is not enriched with Na and that the speculation by Kittrick (1966) that samples C-33 and C-730 might have coatings of sodium aluminate is incorrect.

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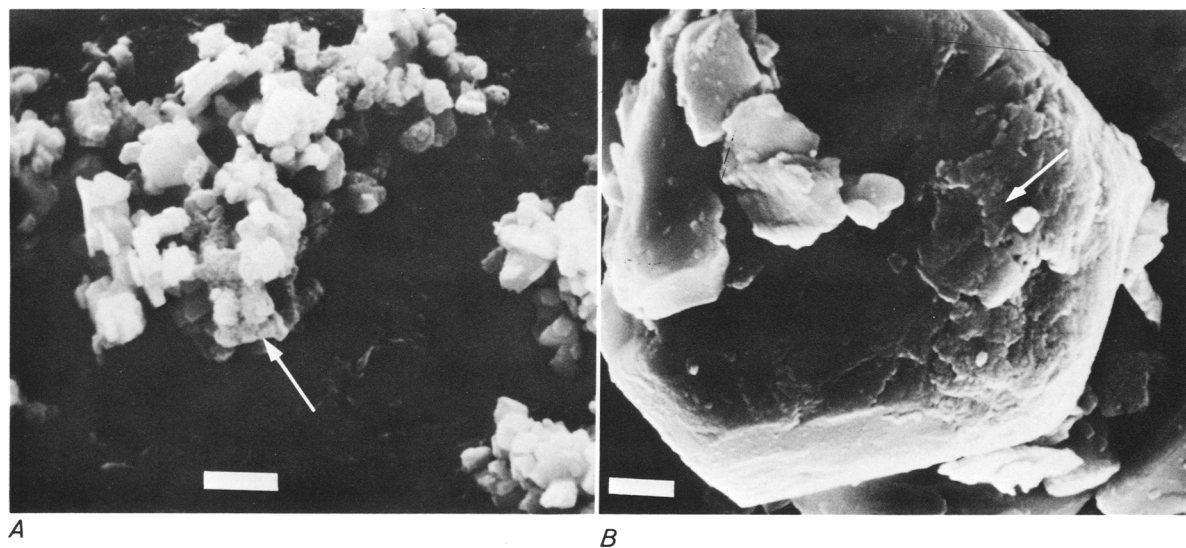


Figure 1. Scanning electron micrographs of untreated gibbsite samples: (A) C-730, (B) FF. The arrows point to poorly ordered material. The white bars represent 1 μm .

Samples C-730, FC, and L contained some well-formed hexagonal tabular crystals (Figure 1) of $<0.1 \mu\text{m}$ to $0.08 \mu\text{m}$ in length (sample C-730) and $>10 \mu\text{m}$ in length (samples FC and L). The crystals of samples FF and L were well-etched by the acid treatment (Figure 2B). The agglomerates in C-33 appeared to be made up of columnar crystals (Figure 2C).

Gibbsite solubility

The length of the runs was, in general, much longer than was necessary for the attainment of equilibrium. With the exception of sample L, the acid-washed gibbsite attained equilibrium from undersaturation in 3 days and from oversaturation in 19 days. Sample L reacted more slowly, but the data (Table 3) suggest that by 228 days this sample was very close to, if not at, equilibrium with the solution. Untreated sample FC attained equilibrium from undersaturation and from oversaturation

at pH 4.3 in one month. The oversaturated sample at pH 3.9 reacted more slowly, but equilibrium was attained by 28 months.

All acid-treated gibbsite samples, except sample L, had the same $*K_{\text{so}}$ (Table 3). The mean $\log *K_{\text{so}}$ at 295°K for samples FF, C-730, and C-33 was 7.71 ± 0.055 . Comparing the two samples of Fisher $\text{Al}(\text{OH})_3$ showed that untreated sample FC (Table 4) had a greater solubility than acid-treated sample FF (Table 3). The mean $\log *K_{\text{so}}$ at 295°K was 8.0 ± 0.03 for sample FC and 7.71 for sample FF. The solubilities at 298°K (Table 5) were calculated from the standard enthalpy of dissolution using the equation:

$$-2.303 \log *K_{\text{so}} = \Delta H^0/RT + S^0/R.$$

The value of ΔH^0 , -88.1 kJ/mole , was calculated from the ΔH^0 for the gibbsite product (Singh, 1972) and the ΔH^0 for the ionization of water (Wagman *et al.*, 1968).

Table 3. Solubility of acid-washed gibbsite samples at 295°K.

Saturation of solution with respect to gibbsite	Sample	3 days		19 days		228 days	
		pH	$\log *K_{\text{so}}^1$	pH	$\log *K_{\text{so}}$	pH	$\log *K_{\text{so}}$
Undersaturated	FF	4.08	7.67	4.10	7.62	4.09	7.74
	C-730	4.10	7.75	4.06	7.67	4.09	7.72
	C-33	4.12	7.80	4.10	7.78	4.09	7.72
Oversaturated	FF	3.92	7.78	3.90	7.62	3.91	7.67
	C-730	3.99	7.92	3.95	7.84	3.88	7.68
	C-33	3.91	7.89	3.89	7.69	3.91	7.64
	L	4.03	8.13	4.03	8.12	4.00	8.02

¹ $*K_{\text{so}} = (\text{Al}^{3+})/(\text{H}^+)^3$.

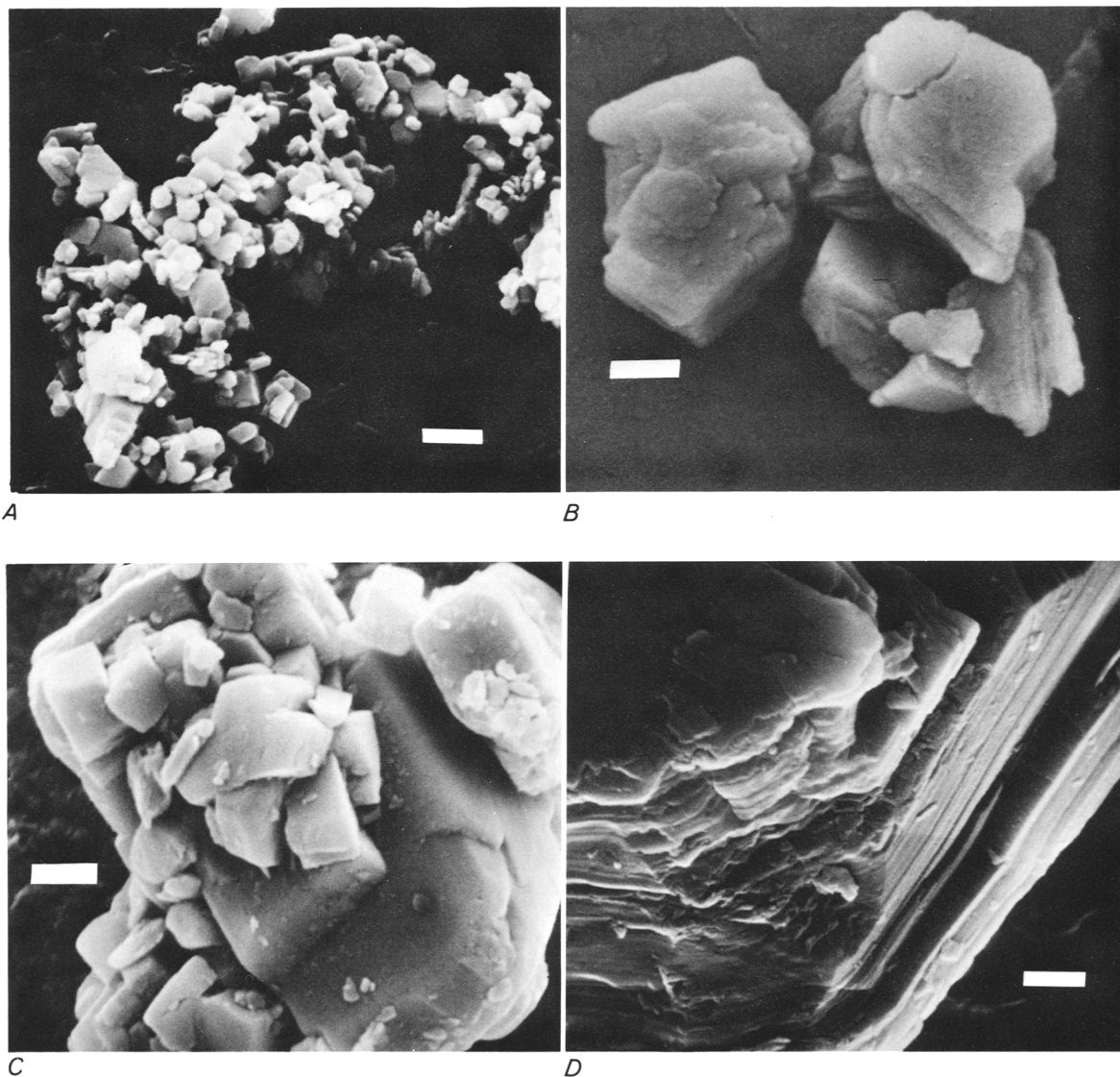


Figure 2. Scanning electron micrographs of acid-washed gibbsite samples: (A) C-730, (B) FF, (C) C-33, (D) L. All except sample L were equilibrated at pH 4 for 228 days. The white bars represent 1 μm .

DISCUSSION

Some laboratory-prepared gibbsites (e.g., sample L) and commercially prepared gibbsites (e.g., samples C-730, C-33, FF, and FC) have complex surface structures that affect the thermodynamic properties. Sample L was prepared by bubbling CO_2 through a hot sodium aluminate solution (Frink and Peech, 1962). This process results in agglomerated crystals that contain 0.26% Na and that have coatings of reactive surface material. The commercial preparations, C-33, FF, and FC, also contained agglomerated crystals, and all samples contained at least 0.2% Na (Table 1). Only a fraction of the Na was associated with reactive surface material. Most

of the relatively large sodium ions were probably located at defects within the crystals which could affect the solubility of the gibbsite. The removal of the reactive surface material by a 14-day contact with 0.1 M HCl significantly decreased the solubility. This solubility decrease is illustrated by comparing the solubility of sample FF (Table 3) with that of sample FC (Table 4). The lower solubility of acid-washed samples L, C-730, and C-33 at 298°K (Table 5) obtained by us compared to the results of Frink and Peech (1962) for untreated sample L and to those of Kittrick (1966) for untreated samples C-730 and C-33 (Table 5) also demonstrates the effect of the acid treatment on solu-

Table 4. Solubility of untreated gibbsite sample FC at 295°K.

Saturation of solution with respect to gibbsite	1 month		7 months		28 months	
	pH	log*K _{so} ¹	pH	log*K _{so}	pH	log*K _{so}
Undersaturated	4.10	8.07	4.10	8.08	4.14	8.08
Oversaturated	3.87	N.D. ²	3.90	8.30	3.86	8.08
	4.29	8.10	4.35	N.D.	4.33	8.14

¹ *K_{so} = (Al³⁺)/(H⁺)³.

² N.D. = not determined.

bility. Other investigators, such as Singh (1972) and May *et al.* (1979), obtained gibbsite solubilities similar to those of Kittrick (1966) (Table 5). The gibbsite used by Singh (1972) (Table 5), however, was precipitated from an acid solution and presumably contained no Na impurity.

Sample L was more soluble than samples C-730, C-33, and FF, even after the acid treatment (Table 5). The greater solubility may be due either to the very finely divided surface material that was not completely removed by the acid treatment (Figure 2D) or to a greater concentration of defects within the gibbsite crystals.

The conditions of formation may be important in determining the solubility of apparently well-crystalline gibbsites. May *et al.* (1979) found that the solubility of a relatively pure natural gibbsite (U = 0.0135%, Zn = 0.0113%, Fe = 0.008%) with a commercial Al(OH)₃ similar to sample FC was four times as soluble as the synthetic gibbsite (Table 5).

The stability in the solubility of sample FC with time (Table 4) demonstrates that the process of Ostwald ripening proceeds extremely slowly in gibbsite suspensions at pH 4. Ostwald ripening should lead to the disappearance of reactive surface material. The progression of the suspensions towards the thermodynamically most stable state was imperceptible even after 20 months (Table 4). The untreated samples of C-730 and C-33 used by Kittrick (1966) remained oversaturated relative to our acid-treated samples (Table 5) even after a four-year equilibration.

In a study of the variation of the solubility of a synthetic gibbsite with pH, May *et al.* (1979) noted a downward shift in solution log(Al) of about 0.5 units with time at pH >6.7. They postulated the formation, in basic solutions, of a more stable phase than gibbsite. The log*K_{so} value of their gibbsite at pH 4 was 0.56 units greater than that for acid-washed samples C-730, C-33, and FF (Table 5). Thus, Ostwald ripening could account for the solubility shift if the ripening process is more rapid in basic pH suspensions. Ostwald ripening could also account for the slow approach to equilibrium in NaOH solutions during the solubility study of Kittrick (1966). In that study, samples which were initially undersaturated with respect to the final solubility mea-

sured, became quickly oversaturated relative to the equilibrium solubility in NaOH and then approached equilibrium from oversaturation. It seems likely that the initial level of Al was controlled by the solubility of the reactive surface material, but with time this material dissolved due to Ostwald ripening. The possibility of more rapid Ostwald ripening, at basic pH values, is also suggested by our observations that gibbsite dissolution is more rapid in basic than in acidic solutions.

The aging of a relatively high-solubility natural gibbsite at pH >6.7 does not lead to solubility values as low as those reported for a synthetic gibbsite aged under the same conditions (May *et al.*, 1979). These differences suggest that internal structural defects rather than reactive surface material account for the differences observed. It seems likely that structural defects may also account for the greater solubility of sample L reported here.

Particle size, or specific surface area, was not a factor in the solubility results (Tables 1, 2, and 5). In the absence of rapid Ostwald ripening, solubility of the untreated samples was controlled by reactive surface material that was not detected by XRD or DTA. Even after acid washing, the samples contained surface particles or individual crystals <0.1 μm in length. The surface energy of these small crystals may influence their solubility. The existence of surface materials also may ac-

Table 5. Gibbsite solubility at 298°K.

Gibbsite sample	Reference	log*K _{so} ¹
C-33, C-730, and FF	Table 3	7.55 ± .055 ²
L	Table 3	7.86 ²
FC	Table 4	7.94 ± .03 ²
C-33, C-730	Kittrick (1966)	7.97 ± .05
L	Frink and Peech (1962)	8.5
Al(OH) ₃ precipitate aged under acidic conditions	Singh (1972)	8.04 ± .03
Commercial purified Al(OH) ₃	May <i>et al.</i> (1979)	8.11
Natural gibbsite	May <i>et al.</i> (1979)	8.7

¹ *K_{so} = (Al³⁺)/(H⁺)³.

² Calculated from solubility at 295°K.

count for the fact that Kittrick (1966) found no difference in the solubility of untreated C-730 and C-33 samples (Table 5). Also, Hemingway *et al.* (1978) reported no particle size effect on the ΔH of solution of commercial gibbsite samples, including samples C-33 and C-730.

The thermodynamically best value for \log^*K_{so} of gibbsite at 298°K is 7.55 ± 0.055 ($pK_{sd} = 34.43 \pm 0.055$), the mean value for samples C-730, C-33, and FF. This value may be somewhat higher than the true solubility of gibbsite due to the Na impurity and the surface energy of finely divided material that may be controlling the solubility.

ACKNOWLEDGMENT

We thank Dr. J. A. Kittrick for supplying two of the gibbsite samples and for reviewing the manuscript.

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(Received 8 June 1981; accepted 17 September 1981)

Резюме—Измерялась растворимость четырех различных образцов гиббсита, включая: два образца производственных уводненных глиноземов, выпускаемых фирмой Алкоа, один образец $Al(OH)_3$ от Фишера и один лабораторный образец. Лабораторно-подготовленные и Алкоа образцы испытывались ранее другими исследователями, но без длительной кислотной обработки. Исследования путем скандирующего электронного микроскопа показали сферический поверхностный материал, который удалялся путем 14-дневной обработки 0,1 М HCl. Растворимость определялась при pH = 4 в условиях пере- и насыщенности с непрерывным возбуждением во время 228 дней. Кислотная обработка коррелировалась с уменьшением величины \log^*K_{so} [$*K_{so} = (Al^{3+})/(H^+)^3$] на около 0,5 единицы. Средняя растворимость трех кислотно-обработанных образцов производственного гиббсита равнялась $\log^*K_{so} = 7,55 \pm 0,555$ ($*K_{so} = 3,5 \times 10^7$). Растворимость кислотно-обработанного лабораторного образца равнялась $\log^*K_{so} = 7,86$ ($*K_{so} = 7,2 \times 10^7$). Большая растворимость лабораторного образца гиббсита приписывается большой концентрации структурных дефектов в кристаллах. [E.C.]

Resümee—Die Löslichkeit von vier unterschiedlich präparierten Gibbsiten wurde gemessen. Es handelte sich dabei um zwei käufliche hydratisierte Al-Oxide, hergestellt von Alcoa, einen Fisher ACS-grade $Al(OH)_3$, und einem Laborpräparat. Die Alcoa Proben und das Laborpräparat wurden schon früher von anderen Autoren untersucht, wobei allerdings keine Langzeitbehandlung mit Säuren durchgeführt wurde. Die Rasterelektronenmikroskopie zeigte kugeliges Oberflächenmaterial, das durch die 14-tägige Behandlung mit 0,1 m HCl entfernt wurde. Die Löslichkeit wurde bei pH 4 sowohl bei Übersättigung als auch bei Untersättigung unter der Einwirkung während 228 Tagen untersucht. Die Säurebehandlung ging mit einer Abnahme des \log^*K_{so} [$*K_{so} = (Al^{3+})/(H^+)^3$] auf etwa 0,5 Einheiten einher. Die Durchschnittslöslichkeit der drei säurebehandelten käuflichen Gibbsitproben betrug $\log^*K_{so} = 7,55 \pm 0,055$ ($*K_{so} = 3,5 \times 10^7$). Die Löslichkeit des säurebehandelten Laborpräparates betrug $\log^*K_{so} = 7,86$ ($*K_{so} = 7,2 \times 10^7$). Die größere Löslichkeit des im Labor hergestellten Gibbsites wird auf eine höhere Konzentration an Fehlstellen im Kristall zurückgeführt. [U.W.]

Résumé—La solubilité de quatre préparations de gibbsite différentes a été mesurée, y compris deux aluminas hydratés commerciaux produits par Alcoa, un $Al(OH)_3$ Fisher grade-ACS, et une préparation de laboratoire. Les échantillons Alcoa et l'échantillon préparé au laboratoire avaient été étudiés précédemment par d'autres investigateurs mais sans utiliser un traitement acide à long-terme. La microscopie électronique balayante a montré du matériel de surface globulaire qui avait été enlevé par un traitement à 0,1 M HCl pendant 14 jours. La solubilité a été déterminée au pH 4 de super- et sous saturation avec une agitation continue pendant 228 jours. Le traitement acide était apparenté à une diminution de \log^*K_{so} [$*K_{so} = (Al^{3+})/(H^+)^3$] d'à peu près 0,5 unités. La solubilité moyenne des trois échantillons de gibbsite commerciaux traités à l'acide était $\log^*K_{so} = 7,55 \pm 0,055$ ($*K_{so} = 3,5 \times 10^7$). La solubilité de la préparation de laboratoire traitée à l'acide était $\log^*K_{so} = 7,86$ ($*K_{so} = 7,2 \times 10^7$). La plus grande solubilité de la gibbsite préparée au laboratoire est attribuée à une plus grande concentration de défauts structuraux dans les cristaux. [D.J.]