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# THE USE OF CAPILLARY SUCTION TIME TO CHARACTERIZE THE SURFACE AREA OF ALUMINUM HYDROXIDE SUSPENSIONS

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## ABSTRACT

The capillary suction time of suspensions of 3 dried aluminum hydroxide powders correlated with the surface area of the powders determined by nitrogen adsorption if the solids content and the aggregation state were controlled. This method is useful for characterizing the surface area of suspended solids as it avoids possible changes in surface area caused by drying the suspension.

## INTRODUCTION

The surface area of a solid suspended in a liquid medium is a very important property of dispersions which has proved to be refractory to measurement. The most widely used method to characterize the surface area of a suspended solid is to dry the suspension to a powder and to determine the surface area of the powder by gas adsorption[1]. There is concern that the drying process can lead to aggregation or particle growth which will affect the surface area measured by gas adsorption. Thus, a method is needed which could be related to the surface area of the suspended solid but which does not require the suspension to be dried.

Capillary suction time refers to the time required for the vehicle in a suspension to travel by capillary forces between the fibers of a filter paper for a standard distance set by two sensing electrodes[2]. Previous studies have shown that the capillary suction time is dependent on the suspended solids content[2] and particle interactions[2,3]. It was believed that the surface area of the suspended solid also influences the capillary suction time. Therefore, a study was undertaken to determine if the capillary suction time could be related to the surface area of a solid dispersed in a liquid medium. Aluminum hydroxide was selected for study because drying is known to affect properties which are related to surface area, such as the rate of acid neutralization[4].

## EXPERIMENTAL

Three samples of dried aluminum hydroxide (Barcroft, Lewes, DE) and one sample of aluminum hydroxide gel (Chattem, Chattanooga, TN) were studied. The equivalent aluminum oxide content was determined by chelatometric titration[5]. The point of zero charge was determined by continuous titration[6]. Capillary suction time measurements were made with a Triton C.S.T. apparatus Type 130 (Triton Electronics, Essex, England) using the 24 mm cylinder and Wickman CST filter paper (Triton Electronics, Essex, England). Surface area measurements on the dried aluminum hydroxide were made using a three point B.E.T. method with nitrogen adsorption (Quantasorb, QS-4, Greenvale, N.Y.). Samples were conditioned by running one adsorption/desorption cycle using 10% v/v nitrogen in helium.

## RESULTS AND DISCUSSION

Aluminum hydroxide has a pH-dependent surface charge[6] which is characterized by the point of zero charge (PZC). Since particle interactions in aluminum hydroxide suspensions have been related to the surface charge, as characterized by (PZC-pH)[6], the effect of pH on the capillary suction time of an aluminum hydroxide gel was determined. The PZC of the sample was found to be 6.75. As seen in Figure 1, the capillary suction time depends on the (PZC-pH) relationship. This is seen most clearly in the suspension containing 9.32% equivalent aluminum oxide (Fig. 1, line A). The lowest value of the capillary suction time (approximately 60 sec.) was observed between pH 6 and 9. This pH region includes the PZC. Thus, it was assumed that the suspension was flocculated in this pH region. The observation that the minimum capillary suction time occurs when the pH is adjusted to the PZC is consistent with the faster filtration rate which is characteristic of flocculated suspensions in comparison to dispersed suspensions[7]. Previous studies of aluminum hydroxide gel have shown that the apparent viscosity[6] is at a maximum and that the diffusion coefficient is at a minimum[8] when the pH is equal to the PZC. Furthermore, these properties were asymmetric about the PZC in a similar fashion as the capillary suction time seen in Figure 1, line A.

The capillary suction time increased when the pH was reduced below pH 6. Lowering the pH causes the surface charge to become increasingly positive according to the following equation[9].

$$\text{Surface Potential} = 59.16 \text{ mV (PZC-pH)}$$

The increased surface charge is expected to lead to a dispersed suspension. A second pH region exhibiting a relatively constant capillary suction time occurs between pH 4.0 and 4.8, i.e., when the suspension is dispersed. Thus, the earlier studies[2,3] were correct in concluding that particle interactions affect capillary suction time. In the present study, the aluminum hydroxide gel had a capillary suction time

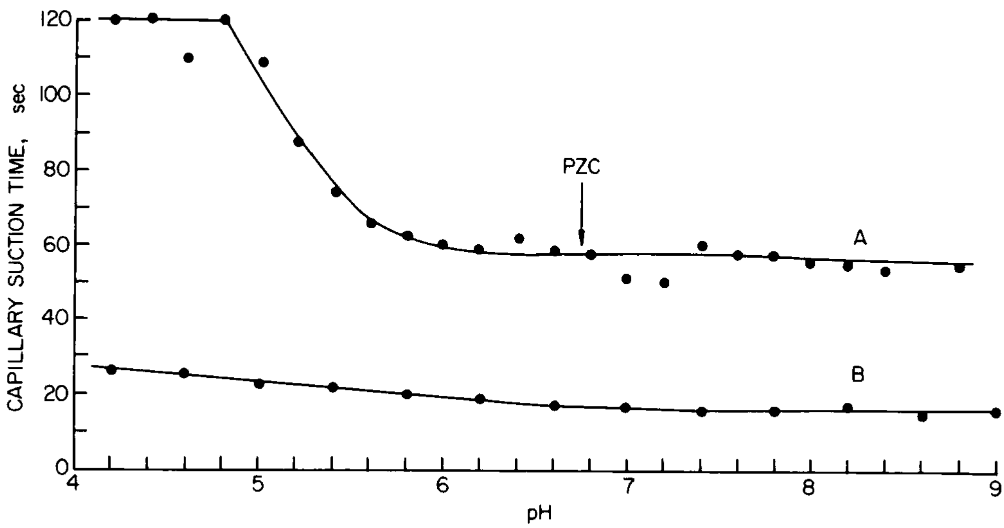


FIGURE 1

Effect of pH on the capillary suction time of an aluminum hydroxide gel containing: A, 9.32% equivalent aluminum oxide; or B, 3.11% equivalent aluminum oxide. The point of zero charge is 6.75.



of approximately 60 sec. when in the flocculated state and a capillary suction time of approximately 120 sec. when in the dispersed state.

The effect of pH on the capillary suction time of the aluminum hydroxide gel was not as apparent when the solids content was reduced to 3.11% equivalent aluminum oxide (Fig. 1, line B). This observation is not surprising as particle interactions in suspensions are directly related to the number of particles in the suspension[10]. The capillary suction time was less for the suspension containing the lower solids content. Baskeville and Gale[2] noted that the solids content of suspensions affected the capillary suction time since for a given volume of filtrate, the thickness of the cake and, thus, its resistance to flow were related to the solids content. Thus, Figure 1 also confirms the earlier conclusion that capillary suction time is dependent on the solids content[2]. Therefore, before capillary suction time can be related to the surface area of the suspended solid, the state of dispersion, i.e., flocculated or dispersed, and the solids content must be defined.

The utility of capillary suction time for characterizing the surface area of a suspended solid was evaluated by examining 3 samples of dried aluminum hydroxide having surface areas of 83, 94 and 106 $m^2/g$ as determined by gas adsorption. The effect of particle interactions was studied first. A suspension was prepared in deionized distilled water by a mechanical stirrer and the pH was adjusted with either 1 N NaOH or 1 N HCl. As seen in Figure 2, the capillary suction time of a 30% w/w suspension of a dried aluminum hydroxide having a surface area of 94 $m^2/g$ was at a minimum between pH 7.4 and 9.4. As noted for the aluminum hydroxide gel studied in Figure 1, this pH region encompasses the point of zero charge. The capillary suction time increased very sharply when the pH was adjusted below 7.0. The dispersed suspension apparently formed a very dense sediment which resisted the outflow of water. Thus, the effect of pH on the capillary suction time for an

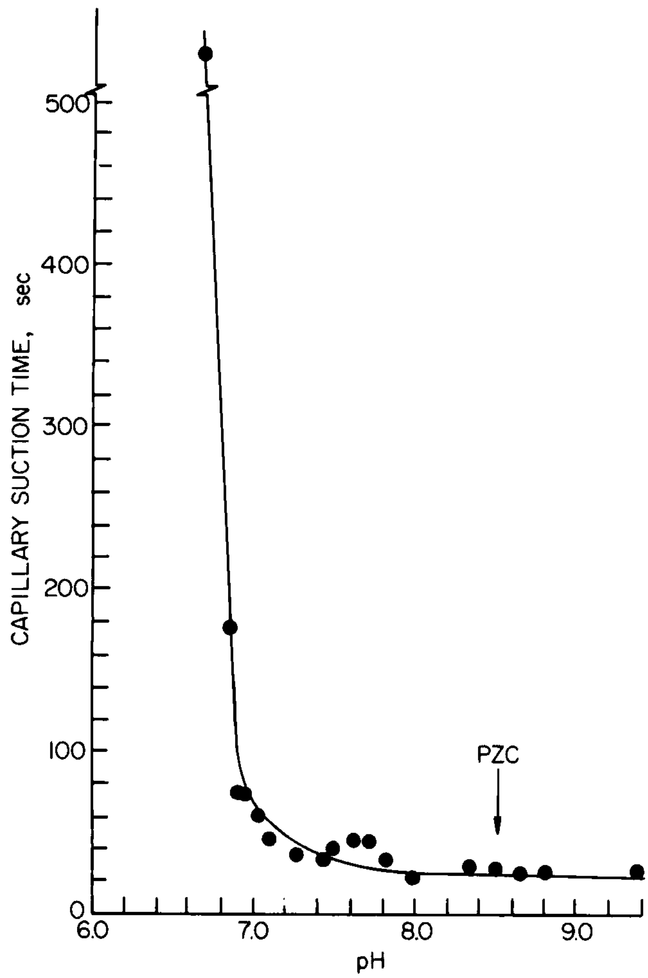


FIGURE 2

Effect of pH on the capillary suction time of a 30% w/w dried aluminum hydroxide suspension. The surface area of the dried aluminum hydroxide was 94 $m^2/g$ and the point of zero charge was 8.45.

aluminum hydroxide gel (Fig. 1) and a suspension of dried aluminum hydroxide (Fig. 2) led to the conclusion that the pH should be adjusted to the PZC in order to obtain reproducible capillary suction time measurements in the shortest period of time.

The effect of hydration time of suspensions of dried aluminum hydroxide on the capillary suction time was also studied. A series of suspensions was prepared containing between 1 and 30% w/w of the dried aluminum hydroxide having a surface area of 94 $m^2/g$. The pH of each suspension was adjusted to 8.45, the PZC. As seen in Table 1, virtually identical capillary suction times were obtained for hydration times of 0.5, 4 or 20 hours. Therefore, 1 hour was chosen as the standard hydration time for all suspensions of dried aluminum hydroxide.

The optimum solids content for using the capillary suction time to characterize the surface area of dried aluminum hydroxide suspensions was determined by preparing suspensions of the 3 samples of dried aluminum hydroxide in which the pH was adjusted to the PZC (Table 2).

The capillary suction times of the three dried aluminum hydroxide suspensions were virtually identical at solids contents up to 20% even though the surface area of the powders ranged from 83 to 106 $m^2/g$. However, the capillary suction times were clearly different for the 3 suspensions at a solids

TABLE 1

Effect of Hydration Time on the Capillary Suction Time of Suspensions of a Dried Aluminum Hydroxide Having a Surface Area of 94 $m^2/g$. The pH Was Adjusted to 8.45, the Point of Zero Charge

| Dried Aluminum Hydroxide % w/w | Capillary Suction Time[1], sec | | |
|---|---|---|---|
| | 0.5 h | 4 h | 20 h |
| 0 | 3.8 (0) | 3.8 (0) | 3.8 (0) |
| 1 | 7.5 (0.6) | 7.1 (0.6) | 8.0 (0.4) |
| 5 | 9.9 (0.1) | 10.0 (0.4) | 8.7 (0.4) |
| 10 | 11.4 (0.1) | 10.6 (0.1) | 11.8 (1.3) |
| 15 | 12.7 (0.9) | 12.7 (0.5) | 12.4 (0.8) |
| 20 | 15.3 (0.1) | 15.0 (0.2) | 14.5 (0.8) |
| 30 | 31.9 (0.4) | 34.9 (0.6) | 34.9 (1.1) |

[1]Mean (standard deviation) of two trials.

content of 30% w/w. In fact, the capillary suction times of the 3 dried aluminum hydroxide suspensions at a solids content of 30% w/w equivalent aluminum oxide were directly related to the surface area of the powder as determined by nitrogen adsorption (Fig. 3). The $R^2$ value for the linear relationship seen in Figure 3 was 0.998. Thus, the capillary suction time of a dried aluminum hydroxide suspension is directly related to

Table 2

Effect of Solids Content on the Capillary Suction Time of Suspensions of Dried Aluminum Hydroxide Having Various Surface Areas. The pH Was Adjusted to the Point of Zero Charge of Each Dried Aluminum Hydroxide.

| Dried Aluminum Hydroxide % w/w | Capillary Suction Time[1], sec | | |
|---|---|---|---|
| | Sample 1[2] | Sample 2[3] | Sample 3[4] |
| 1 | 7.5 (0.8) | 7.5 (0.6) | 6.1 (0.6) |
| 5 | 9.5 (0.6) | 9.9 (0.1) | 10.6 (1.0) |
| 10 | 11.0 (0.2) | 11.4 (0.1) | 10.8 (0.6) |
| 15 | 11.5 (0.7) | 12.7 (0.9) | 11.7 (0.1) |
| 20 | 14.0 (0.7) | 15.3 (0.1) | 13.0 (0.2) |
| 30 | 21.9 (0.2) | 31.9 (0.4) | 45.1 (5.9) |

[1]Mean (standard deviation) of two trials

[2]83 $m^2/g$; pH = PZC = 8.35

[3]94 $m^2/g$; pH = PZC = 8.45

[4]106 $m^2/g$; pH = PZC = 8.43

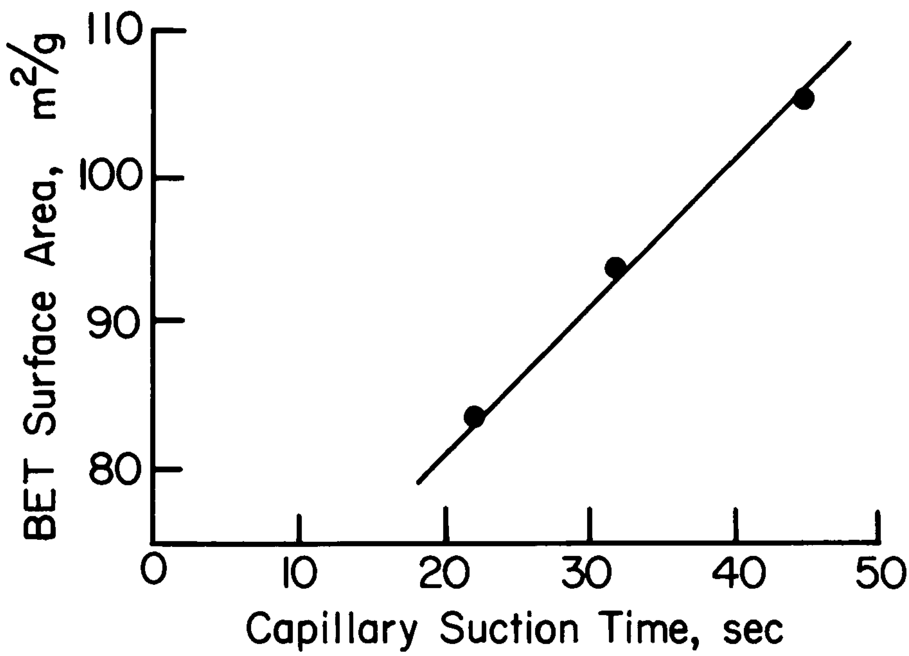


FIGURE 3

Relationship between capillary suction time of 30% w/w suspensions of 3 dried aluminum hydroxide powders and the surface area of the powder determined by gas adsorption. The pH of each suspension was adjusted to the point of zero charge. $R^2 = 0.998$.

the surface area of the powder if the proper solids content and (PZC-pH) relationship are selected.

The capillary suction time may be useful in characterizing the surface area of suspended solids if the solids content and aggregation state are controlled. This method is attractive for application to aluminum hydroxide gel because it avoids changes in the surface area caused by the drying process required for the standard gas adsorption method of determining surface area.


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