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Electrophoretic Separation of Dilute TiO_2 Suspension

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ABSTRACT

A simple apparatus to separate TiO_2 from dilute aqueous suspensions by using electrophoretic principles has been described. Experiments were carried out at different voltages and flow rates. It was observed that voltages lower than 10 V and higher than 50 V were largely ineffectual in separation; 15 V and a 200 mL/h flow rate were established to be the optimum parameters. It was further observed that 96% separation was the maximum obtainable in one stage for this geometry.

INTRODUCTION

Separation of colloidal particles from liquid medium presents special problems because of the inapplicability of a conventional filtration technique. The problem becomes all the more serious when it is necessary to remove colloidal particles from the medium without contamination, e.g., clarification of heavy water used as a moderator and coolant in nuclear reactors (1). The utility of electrokinetic methods in such cases has been discussed by Boyd (2). A similar technique has been used in the field of coal liquification (3) and for the dewatering of petroleum oils (4). Other proposed applications in the same category are in the membrane extraction process (5–7), for water desalination, and for the separation of two phases formed during organic synthesis. In spite of its wide applicability, the mechanism of electrical coalescence is not properly understood even though some attempts have been made to develop mathematical models

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and to derive equations to correlate various parameters (8–10). Solvent extraction is another field where this technique can be applied to increase the efficiency by a factor of 2–3 at the cost of a few watts (11, 12). Two different types of extractors have been described (13). However, this technique has not yet been applied on an industrial scale to any significant extent.

In the field of solid–liquid separations, use of electric fields is of great use where conventional separations like filtration or centrifugation which are restricted to particles with diameters of >10 μm become inapplicable and for smaller particles where costly thermal drying is the only option (e.g., dilute suspensions like latex PVC emulsion or kaolin clay). As the size of the particle becomes smaller, the surface-to-volume ratio increases and it becomes advantageous to use surface-dependent properties like surface charge for separations. It is here that electrophoretic separations have an advantage.

It has been shown that even in cases where a technique like filtration is used, the filter cake produced contains a significant amount of water. Removing this water by conventional methods is costly. For example, the cost of water removal by conventional methods is \$30 and \$16 per ton for latex PVC emulsion and kaolin clay, respectively. Electrofiltration can be applied to these materials at costs of only \$1.81 and \$4 per ton, respectively (14). Even in cases where it is not possible to dewater a slurry to the desired extent, considerable concentration can be achieved which will require minimal secondary drying. This technique has been used for the removal of heavy metals like Cu, Cd, and Pb in wastewater from a clay soil matrix (15).

Various equipments have been used either to overcome some problems or to improve efficiency. Crossflow electrofiltration has been used to remove fine particles suspended in nonaqueous media by applying high voltages (1,000–10,000 V/cm). Freeman (16) used electrofiltration in combination with vacuum to dewater clay. Separation of colloidal α-Al_2O_3 (0.2–0.3 μm) particles from coal and tar sand slurries in tetralin has been reported from synthetic solutions and then repeated for field samples. It was observed that while excellent results were obtained with synthetic samples, field samples did not provide as good separation; this was attributed to different viscosity and other differences (9).

While dewatering can be made efficient by applying an electric field, some limitations have been observed. In studies of heavy particle removal from wastewaters, these limitations include ohmic heating (10, 15), a slow rate of water removal, electrode corrosion, and electrolysis of water.

All the systems described above employ complicated electrode and flow designs to produce a concentrated slurry from a very dilute colloidal solution. So use of a simple assembly would be of great value where the suspended particles have to be removed in situ during the process without introducing any external contaminations. Continuous removal of corrosion products from closed coolant water systems would be one such example. Also when the dilute yet hazardous solutions have to be transported in bulk, this technique would be able to reduce the bulk volume to manageable proportions.

In the present study we selected TiO_2 suspension for electrofiltration studies because TiO_2 easily forms stable suspensions and its high refractive index introduces turbidity in water even at very low concentrations. This high refractive index property makes it very valuable as a filler in polyester textile fibers, in paper, and in the paint industry to impart "whiteness" to the product. The washings from both the factory equipment and floor contain TiO_2 in a dilute turbid suspension. TiO_2 has recently been shown to have unique properties for the adsorption of radionucleides (17), and this can be exploited in the clarification of contaminated heavy water used as a moderator in nuclear reactors. With these in mind, we have carried out experiments on the electrofiltration of TiO_2 (0.35 μm) suspensions in aqueous medium. Separation of TiO_2 from nonaqueous suspensions in the paint and textile industries is also interesting.

EXPERIMENTAL

Apparatus

The apparatus consists (see Fig. 1) of a central graphite rod (0.45 mm diameter and 25 cm in length) used as the anode and eight graphite rods of the same dimensions placed symmetrically along a circle of 30 mm diameter surrounding the anode. These are screwed onto two Perspex sheets so as to make a compact cylindrically shaped assembly of about 30 mm diameter (Fig. 1). This particular arrangement ensures that the horizontal distance of the anode from the cathode is not more than 5 mm at any point. The assembly is inserted into a glass tube in which the inlet and drain are at the bottom and the outlet is 5 cm from the top, in such a way that about a 20-cm length of the electrode assembly dips in the solution. All the outer electrodes are shorted. Both electrodes are connected to a dc voltage source. With this arrangement, the entire length of the liquid receives a uniform electric field.

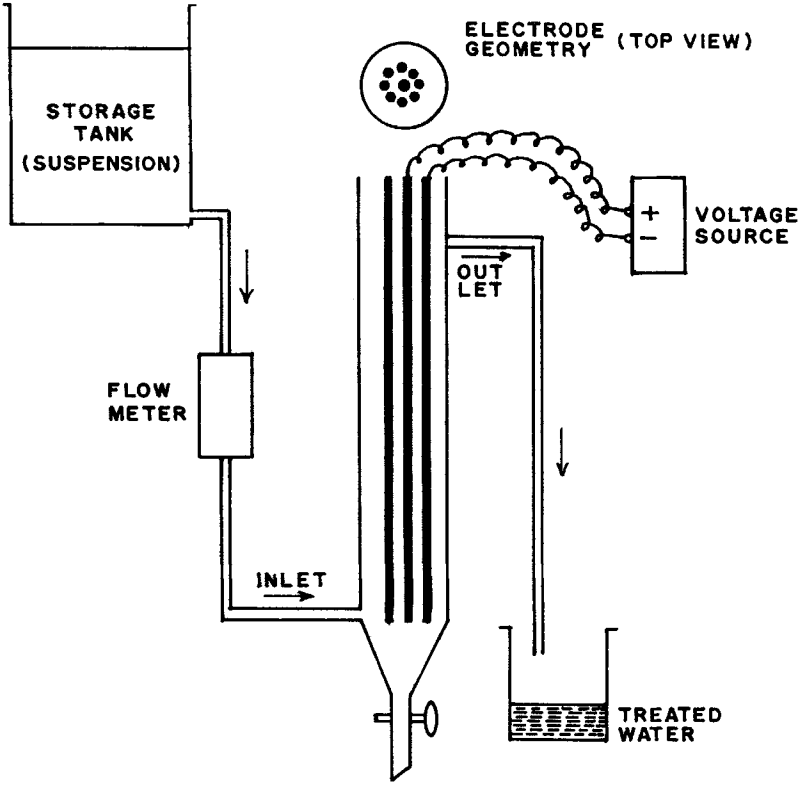

FIG. 1 Vertical cross section of the apparatus.

Preparation of Solution

A.R. grade TiO_2 (E. Merck) was dried at 100°C for 3–4 hours. The average particle size was determined by light-scattering measurement using a Brookhahn BI-90 instrument and found to be ~0.35 μm. A solution of 100 ppm was prepared by mixing a weighed amount of TiO_2 in distilled water (final pH: 6.5). The resulting suspension was quite stable. However, to ensure complete homogeneity, it was sonicated for 1 hour in an ultrasonic tank.

Turbidity was measured on a Urbeco-Hellige Aqua Analyzer-2 at 420 nm wavelength. Different dilutions of this solution were made, and a calibration curve of the percentage transmittance vs concentration was obtained (Fig. 2).

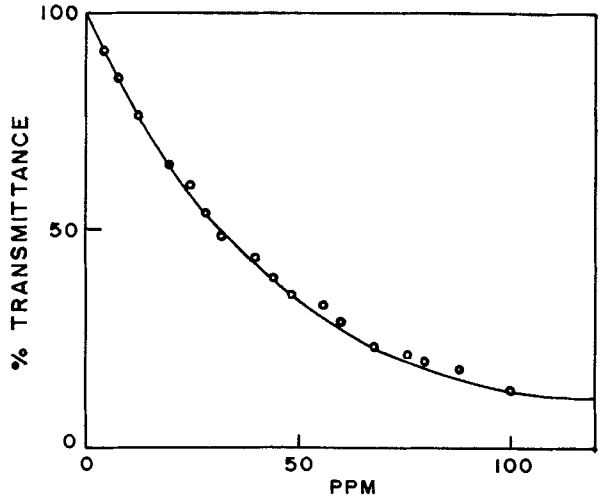

FIG. 2 Calibration curve of turbidity (% transmittance) vs ppm TiO_2.

The solution was stored in the sump (see Fig. 1) and was allowed to flow through the assembly from the bottom inlet at a fixed flow rate. A uniform voltage (dc) was applied to the electrodes after the tube was completely filled with solution. Samples were taken from the top outlet at regular intervals, and the percent transmittance was determined.

RESULTS AND DISCUSSION

Experiments were carried out at flow rates between ~120 and ~450 mL/h and at voltages between 10 and 50 V. It was observed that the TiO_2 particles were attracted toward the anode and deposited on it, implying that they are negatively charged. This was confirmed by reversing the voltage. The particles always deposited on the anode. Beyond 50 V, evolution of gases posed a serious problem as it introduced a turbulence which hampered separation. Tables 1, 2, and 3 give a summary of experimental results. The effect of flow rate and the voltage are separately shown in Figs. 3 and 4. It can be seen that the initial rate of separation is independent of the flow rate but depends on the voltage rate, increasing with an increase in voltage. It can further be seen that a maximum clearance of 90% transmittance, corresponding to about 96% separation, is obtained. This means that out of 100 ppm of TiO_2, 4 ppm remains in the outlet liquid.

TABLE 1

10 V			15 V			30 V			30 V	
Time (min)	266 mL/h, % transmittance	118 mL/h, % transmittance	Time (min)	200 mL/h, % transmittance	324 mL/h, % transmittance	Time (min)	360 mL/h, % transmittance	288 mL/h, % transmittance	Time (min)	129 mL/h, % transmittance
0	13	13	0	13	36	0	13	47	0	13
15	38	44	15	69	45	15	47	54	15	67
25	45	48	25	75	59	20	53	66	25	69
35	37	61	35	76	57	25	56	69	35	69
45	43	58	45	77	60	30	60	70	45	76
55	48	47	55	81	58	35	66	74	55	82
65	49	38	65	79	56	40	64	74	65	86
75	51	61	75	82	59	45	64	74	75	87
85	54	61	85	86	59	50	64	73	85	87
95	51	61	95	86	58	60	66	76	95	87
105	51	61	105	86	59	65	67	77	105	87
115	50	61	115	86	59	75	69	74		
						85	72	76		
						95	70	75		
						105	68	76		
						115	68	75		
						[a]	87			

[a] After being static for 180 minutes (after 105 minutes of experiment).

TABLE 2

40 V				50 V		50 V	
Time (minutes)	128 mL/h, % transmittance	190 mL/h, % transmittance	288 mL/h, % transmittance	Time (minutes)	110 mL/h, % transmittance	Time (minutes)[a]	343 mL/h, % transmittance
0	13	13	13	0	13	0	13
15	65	66	64	15	67	5	32
25	77	66	75	25	75	10	48
35	81	81	83	35	90	15	59
45	83	87	84	45	91	25	65
55	86	90	82	55		30	73
65	86	90	80	65		35	77
75	86	90	79	75		40	80
85	86	90	81	85		45	80
95	86	90–91	78	95		50	80
105	86	90	78	105		55	80
						60	80

[a] Electrode voltage fluctuation very serious after 30 Minutes. Significant rise in solution temperature.

TABLE 3
Calibration (concentration vs turbidity) for Various Volumes of 0.01% (100 ppm) Solutions Diluted to 25 mL

No.	Dilution	Concentration, ppm	% Transmittance
1	1–25	4	92
2	2–25	8	85
3	3–25	12	77
4	5–25	20	65
5	6–25	24	61
6	7–25	28	54
7	8–25	32	49
8	10–25	40	44
9	11–25	44	39
10	12–25	48	35
11	13–25	56	33
12	15–25	60	29
13	17–25	68	24
14	19–25	76	22
15	20–25	80	20
16	22–25	88	17
	Original	100	13

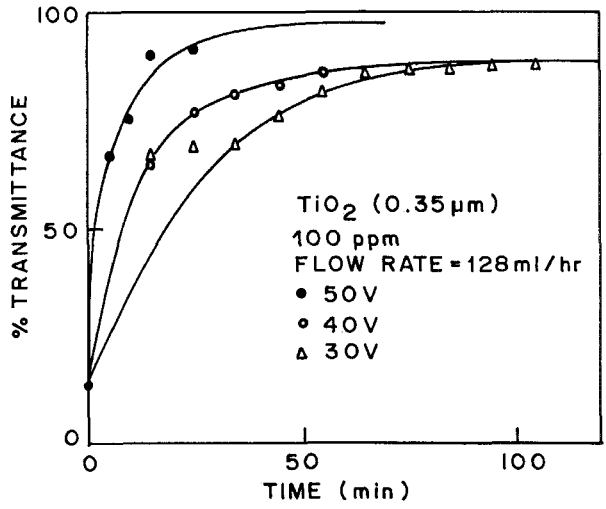

FIG. 3 Plot of % transmittance vs time at constant flow rate and different voltages.

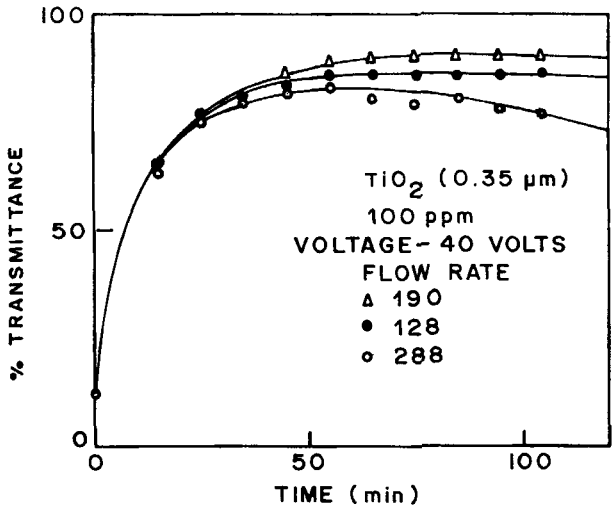

FIG. 4 Plot of % transmittance vs time at constant voltage (40 V) and different flow rates.

This seems to be the limit for this one-stage geometry. If greater filtration is required, then a second stage or a different geometry of the electrode will have to be employed. The plots further reveal that steady state is obtained at medium to high voltage (30–50 V) after about 35–40 minutes. This corresponds to particle velocities in the 10^{-3} cm/s range depending on the flow rate. This is the time taken for the liquid from the bottom to reach the overflow level. It can further be seen that at high voltages, the final separation efficiency is by and large independent of the flow rate. Table 1 suggests that 15 V and 200 mL/h are the optimum conditions under which steady state is obtained within 15–20 minutes. This could be due to the fact that at high voltages the turbulence set up by gases evolving at the cathode adversely affect the separation (over and above heating of the solution). At low voltage the electric field is not sufficiently strong to effect separation. A medium voltage of 15 V seems to be an ideal compromise.

The experiments were carried out under the optimum conditions for prolonged periods, and no noticeable deterioration in electrode conditions or significant gas evolution resulting from the turbulence was noticed. Further, if a thick slurry amounting to 25–30 mL is taken out after every 3–4 hours of operation, by drain, it is possible to continue the operation almost indefinitely. Light-scattering experiments showed that the particle

size of the thick slurry obtained after prolonged experiment did not materially differ from the original particle size (0.35 μm original size, 0.45 μm final size).

SUMMARY

An electrophoretic separation of TiO_2 slurries in aqueous medium is described. It is shown that by using voltages of the order of 10–50 V, about 96% separation efficiency can be obtained in continuously flowing systems. The effects of voltage and flow rate were investigated.

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