WATER RESEARCH 45 (2011) 6301–6307

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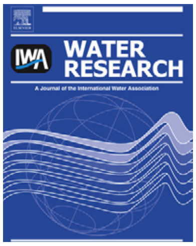

Using magnetic seeds to improve the aggregation and precipitation of nanoparticles from backside grinding wastewater

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ARTICLE INFO

Article history:
Received 29 December 2010
Received in revised form
29 April 2011
Accepted 31 August 2011
Available online 29 September 2011

Keywords:
Backside grinding (BG)
Silica nanoparticles
Magnetic seeds
Applied magnetic field
Aggregation

ABSTRACT

Backside grinding (BG) wastewater treatment typically requires large quantities of chemicals, i.e. polyaluminum chloride (PAC) coagulant and produces considerable amounts of sludge, increasing the loading and cost of subsequent sludge treatment and disposal processes. This study investigated the effects of the addition of magnetic seeds ($FeO^*Fe_2O_3$) of selected particle sizes and of optimized combinations of magnetic seeds and PAC on the aggregation of silica nanoparticles from BG wastewater and on the sedimentation time at various pH values (5–9). The results show that the turbidity of BG wastewater was significantly reduced by the magnetic aggregation treatment. The dosage of PAC combined with 2.49 $g\,L^{-1}$ or 1.24 $g\,L^{-1}$ of magnetic seeds was reduced by 83% (from 60 to 10 $mg\,L^{-1}$) compared to the conventional process of using only PAC as a coagulant. The turbidity of the BG wastewater, initially 1900–2500 NTU, could also be successfully decreased about to 23 NTU by the addition of 3.74 $g\,L^{-1}$ magnetite ($FeO^*Fe_2O_3$) only at pH 5 with an applied magnetic field of 1000 G. Different coagulation conditions using magnetic seeds combined with coagulant resulted in different aggregation performances. The treatment performance was more effective by using two-stage dosing, in which magnetic seeds and PAC were added separately, than that with one-stage dosing, where the magnetic seeds and PAC were added simultaneously during rapid mixing. The two-stage dosing allowed for a reduction in the optimum dosage of magnetic seeds from 3.74 $g\,L^{-1}$ to 2.49 $g\,L^{-1}$ or 1.24 $g\,L^{-1}$ without affecting performance when coupled with 0.01 $g\,L^{-1}$ of PAC coagulant. The developed method effectively reduced the production of waste sludge.

1. Introduction

Backside grinders (BG), as used in the semiconductor industry, produce turbid wastewater containing substantial quantities of fine abrasive particles. Current methods for the purification of BG wastewater employ coagulation and sedimentation to remove fine particles. But these methods not only require large quantities of chemicals but also produce a considerable amount of sludge, increasing the cost of subsequent sludge treatment and disposal processes. The clarification of BG

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doi:10.1016/j.watres.2011.08.067

wastewater using such methods is difficult because the suspended particles exhibit high stability and resistance to diffusion (Matteson et al., 1995; Touris et al., 2001). In general, semiconductor foundries use coagulants to aggregate wastewater nanoparticles, forming flocs to facilitate sedimentation prior to filtration (e.g., sand or membrane filtration). However, the lifespan of these filtration devices is short due to membrane pore blockage. Thus, the use of these membrane technologies is ineffective (Weigert et al., 1999; Yang and Tsai, 2006; Lin and Yang, 2004).

There have been recent reports of the successful application of aggregation technologies employing magnetic seeds. Magnetic separation has attracted great attention because the magnetic force is a long-range attraction, and thus enhances the removal of waste nanoparticles. The main advantage of this technology is that it can treat a mass of wastewater in a very short period of time and produces no contaminants, and reduces the quantity of chemical waste sludge. As a result, it has been widely used in the textile industry, the field of biology and in environmental protection (Chin et al., 2006; Rocher et al., 2008; Lo et al., 2008; Pal and Alocilja, 2009). Higashitani et al. (1996) investigated the effects of magnetic fields on the stability of nonmagnetic colloidal particles and suggested that colloidal stability is influenced by magnetic fields through alteration of the structure of molecular water clusters and ions, either adsorbed on the particle surface or within the medium (Kney and Parsons, 2006). Raw water processing normally involves physicochemical procedures based on the coagulation and flocculation of suspended solids (SS) and colloids and the adsorption of soluble material onto solid substrates such as metal hydroxides (Bolto and Gregory, 2007). Using the jar test system, magnetic seeds have been shown to successfully bind and precipitate phosphate from wastewater; adjusting the pH and calcium concentration can improve the sedimentation efficiency (Karapinar et al., 2004, 2006). Magnetic composites can adsorb contaminants from aqueous or gaseous effluents and can then be easily separated from the medium by a simple magnetic process (Oliveira et al., 2004). Highly turbid raw water has been effectively treated using $FeO^*Fe_2O_3$ magnetic particles, reducing the turbidity from 9600 to 20 NTU (Lo et al., 2008). In another study, magnetite nanoparticles were synthesized and used as seed particles. The turbidity of the CMP wastewater was reduced from 110 NTU to 7 NTU at a solution pH of 6 with no salt addition (Chin et al., 2006).

The current study was divided into three stages. The first stage investigated magnetic seed formulations and their characterization. The second stage studied the extent of magnetic seeding-induced aggregation in BG wastewater under different physical and chemical conditions, namely, magnetic seed dosage, pH, applied magnetic field, and the use of coagulants. In this stage, coagulants were added to increase the collision frequency and aggregation among the magnetic seeds and nanoparticles, thereby reducing the amount of magnetic seeds required for similar turbidity removal efficiency with the same sedimentation time. The third stage investigated the effect of the magnetic seeding aggregation technique on treatment efficiency.

2. Materials and methods

2.1. Samples

Wastewater samples were obtained from semiconductor companies. The wastewater from the silicon wafer backside grinding process is mainly composed of fine nanoparticles with highly stable silica particles (SiO_2) and some trace metals that originate from rinsing silicon wafers with ultrapure water.

2.2. Preparation and characterization of magnetic seeds

Various dosages of magnetic seeds were prepared by combining $FeCl_3 \cdot 6H_2O$ (ferric chloride, Shimakyu, Japan) and $FeCl_2 \cdot 4H_2O$ (ferrous chloride tetrahydrate, Hanawa, Japan), followed by titration with $NaOH_{(aq)}$ (see Table 1) with stirring at 300 rpm, at room temperature. The optimum molar ratio of Fe^{2+} to Fe^{3+} was taken as 2:3, as this ratio has been shown to produce a synergistic effect (Hu et al., 2005). The prepared mixture was black. Finally, the magnetic seeds were washed with deionized water. When the aggregation reaction was completed, there were still impurities remaining in the colloidal water suspension ($pH = 5.5 \pm 0.5$). The dried magnetic seeds were analyzed for particle size and distribution by X-ray powder diffraction, and the results are shown in Fig. 1(a) and (b). The particle size of the magnetic seeds in the slurry was approximately 100–300 nm, the same size as reported by Chin et al. (2006). The diffraction angles of the magnetic seed crystals appeared at approximately $2\theta = 30°, 35°, 43°, 57°$ and $63°$. The chemical composition of the magnetic seeds was confirmed to be $FeO^*Fe_2O_3$ by comparison of this diffraction pattern with previously reported patterns (Deng et al., 2003) found in the database of the Joint Committee on Powder Diffraction Standards (JCPDS). Finally, 100–300 nm

Table 1 – Preparation of different magnetic seed dosages.

No.	$FeCl_2 \cdot 4H_2O$ ($g\,L^{-1}$)	$FeCl_3 \cdot 6H_2O$ ($g\,L^{-1}$)	NaOH ($g\,L^{-1}$)	Molar ratio of Fe^{2+}:Fe^{3+}	Theoretical dosages ($g\,L^{-1}$)
1	1.24	2.5	1.62	2:3	1.24
2	2.47	5.0	3.23	2:3	2.49
3	3.70	7.5	4.84	2:3	3.74
4	4.93	10.0	6.45	2:3	4.99
5	6.16	12.5	8.06	2:3	6.23
6	7.39	15.0	9.67	2:3	7.49
7	8.62	17.5	11.28	2:3	8.72