Separation of Cerium Oxide Abrasive and Glass Powder in an Abrasive-Glass Polishing Waste by Means of Liquid–Liquid–Powder Extraction Method for Recovery: A Comparison of Using a Cationic and an Anionic Surfactant Collector

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Abstract: Applying a cerium oxide abrasive to polish glass components generates a polishing waste containing the cerium oxide abrasive and the glass powder produced during polishing. This research applied the liquid–liquid–powder extraction method to separate the cerium oxide abrasive and the polished glass powder in an abrasive-glass polishing waste for recovering the cerium oxide abrasive. Two liquids of isooctane and water were utilized. The effectiveness of using a cationic and an anionic surfactant collector, i.e., dodecylamine acetate (DAA) and sodium oleate (NaOL), respectively, in improving their extraction and separation was investigated and compared. The results indicated that NaOL addition could improve the mutual separation of cerium oxide abrasive and glass powder but DAA could not, because the former could selectively improve the extraction of cerium oxide abrasive from the water phase to isooctane phase whereas the latter could improve that of both powders. Optimal separation for the cerium oxide abrasive and the polished glass powder in an abrasive-glass polishing waste were achieved by adding NaOL of 7.5 kg/ton at pH 7; the content of cerium oxide abrasive in the solid recovered from the isooctane phase was 96.4% with a recovery of 88.1%.

Keywords: cerium oxide abrasive; glass powder; separation; recovery; liquid–liquid–powder extraction; surfactant collector; dodecylamine acetate (DAA); sodium oleate (NaOL)

1. Introduction

The surfaces of glass components such as optical glass and panel glass need to be precisely polished before they can be used for fabricating the optical lenses of cameras or the glass substrates of liquid crystal displays. A cerium oxide abrasive is often used for polishing the glass components [1,2]. The polishing process generates a solid waste containing the cerium oxide abrasive and the polished glass. Currently, a major portion of the polishing waste is discarded in landfills. However, the cerium
oxide abrasive should ideally be recovered from this polishing waste, not only for reducing the production of solid waste but also to act as a secondary source of rare earth elements.

Several methods have been developed for recovering the rare earth elements in abrasive-glass polishing waste [3–8]. Most of such methods are based on pyrometallurgy [3,4] or hydrometallurgy [5–8]. Although technically feasible, both types of method have some limitations such as the consumption of high energy in the pyrometallurgical process and the requirement of wastewater treatment in the hydrometallurgical process. Substantially, an abrasive-glass polishing waste mainly comprises two powder types: cerium oxide abrasive and glass powder. Effectively separating these two powders can be beneficial for subsequent rare earth element metallurgical processes such as reducing reagent requirement and wastewater production. Moreover, the recovered cerium oxide abrasive can be used for polishing again if there is no significant change in its particle size distribution [8]. However, it is difficult to separate the cerium oxide abrasive and the polished glass by applying common mineral processing methods such as froth flotation or gravity separation because the particle sizes of the two powders are both smaller than 1 μm [1,2].

The liquid–liquid–powder extraction method is a method similar to froth flotation, which applies the difference in surface property to separate a powder mixture [9–13]. A polar liquid and a nonpolar liquid (usually water and an organic solvent) are used to create two immiscible liquid phases. Hydrophobic powders attach to the droplets of nonpolar liquid and move to the phase of nonpolar liquid or to the interface between the two liquids, whereas hydrophilic powders do not attach to the droplets of nonpolar liquid and stay in the phase of polar liquid. Compared with the froth flotation method, the liquid–liquid–powder extraction method is more effective for separating ultrafine powders of less than 10 μm [14,15]. This is because the droplets of nonpolar liquid have a smaller size but higher stability than air bubbles, which can increase the probability of their collision and contact with ultrafine powders [14,15]. Moreover, similar to the froth flotation method, the addition of a surfactant collector is usually needed and important in the liquid–liquid–powder extraction method to selectively adjust the oil wettability of part of the powders to improve their extraction and separation. The liquid–liquid–powder extraction method has been applied to separate ultrafine powders for mineral processing and material recycling [11–13,15–19].

This research applied the liquid–liquid–powder extraction method to separate the cerium oxide abrasive and the polished glass powder in an abrasive-glass polishing waste for recovering the cerium oxide abrasive. Two liquids of isooctane and water were utilized. The effectiveness of using a cationic and an anionic surfactant collector, i.e., dodecylamine acetate (DAA) and sodium oleate (NaOL), respectively, on improving the extraction and separation was investigated and compared. Firstly, the extracted fraction (i.e., the mass percentage of the powder recovered in the isooctane phase) of the cerium oxide abrasive and the glass powder without and with the addition of each surfactant collector was investigated separately to observe the extraction behavior of each powder. Subsequently, the separation of the cerium oxide abrasive and the polished glass powder in an abrasive-glass polishing waste was performed to decide on the optimal separation conditions, and the separation results were compared. Finally, the mechanism of each surfactant collector addition to improve the extraction and the separation of these two powders was discussed.

2. Materials and Methods

Three experimental materials—cerium oxide abrasive, abrasive-glass polishing waste, and glass panel—were provided by Global Display Taiwan Co., Ltd. The cerium oxide abrasive contained 70 wt% CeO₂ which had a mean particle size of \( D_{50} = 0.482 \mu m \), with \( D_{10} = 0.105 \mu m \) and \( D_{90} = 2.066 \mu m \). The abrasive-glass polishing waste was produced by applying the cerium oxide abrasive to polish the glass panel. After the polishing process, flocculants were not added into the polishing waste for dewatering, and therefore the polishing waste only contained the cerium oxide abrasive and the polished glass powder. The content of cerium oxide abrasive and polished glass powder in the as-received polishing waste sample was 80.5 and 19.5 wt%, respectively. The polishing waste had
a mean particle size of $D_{50} = 0.571 \, \mu m$, with $D_{10} = 0.329 \, \mu m$ and $D_{90} = 1.180 \, \mu m$. The glass panel was crushed and ground using a stainless-steel mortar and an agate mortar autogrinder (ANM1000, Nittokagaku Co., Ltd., Tokyo, Japan), respectively, to produce a glass powder. The glass powder was further ground using a bead mill (PML-2, Bühler Ltd., Uzwil, Switzerland) to a mean particle size of $D_{50} = 0.609 \, \mu m$, with $D_{10} = 0.099 \, \mu m$ and $D_{90} = 3.901 \, \mu m$, to simulate the polished glass powder in the polishing waste. The above particle size distribution results of all powder samples were analyzed by a laser particle size analyzer (Bettersizer S2, Bettersize Instruments Ltd., Dandong, China).

Two liquids of isooctane (purity: 99.5%) (Sigma-Aldrich Co., St Louis, MO, USA) and distilled water were utilized. DAA (C$_{14}$H$_{31}$NO$_{2}$, purity: 97%) (Tokyo Chemical Industry Co., Ltd., Tokyo, Japan) and NaOL (C$_{17}$H$_{33}$COONa, purity: 98.5%) (Tokyo Chemical Industry) were utilized as the cationic and the anionic surfactant collector, respectively. The reagent grades of 1 M hydrochloric acid solution (Sigma-Aldrich) and 1 M sodium hydroxide solution (Sigma-Aldrich) were utilized to adjust the solution pH.

To investigate the individual extracted fraction of the cerium oxide abrasive and the glass powder, a decided amount of cerium oxide abrasive and glass powder was put into a beaker with 80 mL of distilled water and then dispersed ultrasonically. The powder solution was poured into a Pyrex separatory funnel after adjusting its pH to the desired value. Subsequently, the surfactant collector was added into the powder solution followed by shaking the separatory funnel mechanically for 15 min for conditioning. After the introduction of 20 mL of isooctane, the separatory funnel was shaken for an additional 15 min and then kept still for 30 min to enable the formation of the isooctane phase and the water phase. The two liquid phases were drawn out into individual beakers. After centrifugation and drying, the obtained solids were weighed to decide the extracted fraction (i.e., the mass percentage of the powder recovered in the isooctane phase) of individual powders. Unless otherwise noted, the volumes of isooctane and distilled water were maintained at 20 and 80 mL, respectively.

To evaluate the mutual separation of the cerium oxide abrasive and the polished glass powder, the aforementioned experimental procedure was applied to the as-received abrasive-glass polishing waste sample. The solid recovered from the isooctane phase and the water phase was weighed. The recovered solid was then dissolved using aqua regia, and the amount of cerium oxide abrasive in the solid was analyzed by an inductively coupled plasma optical emission spectrometer (ICP–OES, Optima 8300, Perkin Elmer, Waltham, MA, USA). The separation results were demonstrated by the content and the recovery of cerium oxide abrasive in the solid recovered from the isooctane phase, calculated as follows.

\[
\text{Content (\%) = \frac{\text{The mass of cerium oxide abrasive in the recovered solid (g)}}{\text{The mass of the recovered solid (g)}} \times 100\%}
\]

\[
\text{Recovery (\%) = \frac{\text{The mass of cerium oxide abrasive in the recovered solid (g)}}{\text{The mass of cerium oxide abrasive in the polishing waste (g)}} \times 100\%}
\]

Each experiment was performed at least twice. The mean value and the standard deviation were calculated and presented.

3. Results

3.1. Extraction of the Cerium Oxide Abrasive and the Glass Powder

3.1.1. No Surfactant Collector Addition

Figure 1 presents the extracted fraction from water to isooctane phase of the cerium oxide abrasive and the glass powder as a function of pH without surfactant collector addition. When a surfactant collector was not added, the extracted fraction of either the cerium oxide abrasive or the glass powder was less than 10% across the pH range of 3–11, indicating that most of the cerium oxide abrasive and the glass powder remained in the water phase. The results suggest that the surface properties of...
the two powders are both hydrophilic. In order to improve their extraction to achieve ideal mutual separation for recovery, the addition of a surfactant collector is needed.

![Figure 1](image1.png)

**Figure 1.** Extracted fraction (mass percentage of the powder recovered in isooctane phase) of the cerium oxide abrasive and the glass powder as a function of pH without surfactant collector addition (Experimental conditions: cerium oxide abrasive/glass powder ca. 1 g, isooctane 20 mL, water 80 mL).

### 3.1.2. DAA Addition

Figure 2 shows the extracted fraction from water to isooctane phase of the cerium oxide abrasive and the glass powder as a function of pH with dodecylamine acetate (DAA) addition. The DAA dosage was given as kilograms of DAA added per ton of powder. When DAA was added, the extracted fraction of the cerium oxide abrasive seldom increased at pH 3–5 but significantly increased above pH 5. Higher extracted fraction was obtained at pH 7–9 together with higher DAA dosage. An extracted fraction of nearly reached 100% was obtained at pH 9 when 0.5 kg/ton DAA was added. At pH 11, the extracted fraction of the cerium oxide abrasive decreased. On the other hand, the extracted fraction of glass powder substantially increased at pH 3–9, but also decreased at pH 11. As the DAA dosage increased, the extracted fraction of the glass powder also increased. An extracted fraction that nearly reached 100% was obtained at pH 5–9 when 0.5 kg/ton DAA was added. The results suggested that DAA addition could improve the extraction of cerium oxide abrasive to the isooctane phase at pH 7–9, and that of glass powder at pH 3–9. In addition, the higher DAA dosage, the higher the extracted fraction that could be obtained.

![Figure 2](image2.png)

**Figure 2.** Extracted fraction (mass percentage of the powder recovered in isooctane phase) of the cerium oxide abrasive and the glass powder as a function of pH with dodecylamine acetate (DAA) addition (Experimental conditions: cerium oxide abrasive/glass powder ca. 1 g, isooctane 20 mL, water 80 mL).
3.1.3. NaOL Addition

Figure 3 illustrates the extracted fraction from water to the isooctane phase of the cerium oxide abrasive and the glass powder as a function of pH with NaOL addition. The NaOL dosage was given as kilograms of NaOL added per ton of powder. When NaOL was added, the extracted fraction of cerium oxide abrasive substantially increased in the tested pH range of 3–11. As the NaOL dosage increased, the extracted fraction of cerium oxide abrasive also increased. A higher extracted fraction of cerium oxide abrasive (∼98%) was obtained at pH 7 with a NaOL dosage of 2 kg/ton. Decreasing or increasing the pH from 7 caused a reduction in the extracted fraction. By contrast, the extracted fraction of glass powder showed no considerable change with NaOL addition, which was ca. 10% across the pH range being tested. In addition, the increase in NaOL dosage could not improve the extracted fraction. The results suggested that NaOL addition could improve the extraction of cerium oxide abrasive throughout the pH, especially at pH 7, but could not improve that of glass powder.

![Graph showing extracted fraction vs pH](image)

**Figure 3.** Extracted fraction (mass percentage of the powder recovered in isooctane phase) of the cerium oxide abrasive and the glass powder as a function of pH with NaOL addition (Experimental conditions: cerium oxide abrasive/glass powder ca. 1 g, isooctane 20 mL, water 80 mL).

As an example of the above extraction experiment, Figure 4 shows photographs of the cerium oxide abrasive and the glass powder as observed after the application of the liquid–liquid–powder extraction method along with a 2 kg/ton NaOL addition at pH 7. Clearly, nearly all of the cerium oxide abrasive was extracted to the isooctane phase. By contrast, most of the glass powder stayed in the water phase.

![Photographs of separatory funnel](image)

**Figure 4.** Photographs of separatory funnel of (a) cerium oxide abrasive, (b) glass powder after the application of liquid–liquid–powder extraction with 2 kg/ton NaOL addition at pH 7 (Experimental conditions: cerium oxide abrasive/glass powder ca. 1 g, isooctane 20 mL, water 80 mL).
3.2. Separation of the Cerium Oxide Abrasive and the Glass Powder in the Abrasive—Glass Polishing Waste

3.2.1. No Surfactant Collector Addition

Figure 5 presents the content and recovery of cerium oxide abrasive in the solid recovered from the isooctane phase as a function of pH without surfactant collector addition. When the surfactant collector was not added, the content and recovery of cerium oxide abrasive had no significant change throughout the tested pH, ranging from 3 to 11. For each pH, the content of cerium oxide abrasive was ca. 80–90%, which was slightly higher than its initial content (80.5%) in the as-received polishing waste. However, the recovery of cerium oxide abrasive was ca. 10%, indicating that most of the cerium oxide abrasive stayed in the water phase without being extracted to the isooctane phase. The results suggested that the cerium oxide abrasive and the glass powder could not be well separated without surfactant collector addition.

![Figure 5](image_url)

**Figure 5.** The content and recovery of cerium oxide abrasive in the solid recovered from the isooctane phase as a function of pH without surfactant collector addition (Experimental conditions: abrasive—glass polishing waste ca. 2 g, isooctane 20 mL, water 80 mL).

3.2.2. DAA Addition

Figure 6 shows the content and recovery of cerium oxide abrasive in the solid recovered from the isooctane phase as a function of pH with DAA addition. When adding 0.5 kg/ton DAA, the content of cerium oxide abrasive in the solid recovered from the isooctane phase was ca. 65–80% in the experimental pH range from 3 to 11, which was below the its initial content (80.5%) in the as-received polishing waste sample. On the contrary, the recovery of cerium oxide abrasive in the solid recovered from the isooctane phase seldom increased at pH 3–5 but significantly increased when the pH was higher than 5. More than 90% recovery was obtained at pH 7–9. At pH 11, the recovery significantly decreased.

Although a high recovery of cerium oxide abrasive in the solid recovered from the isooctane phase could be obtained after DAA addition at pH 7–9, the content of cerium oxide abrasive was below the initial one, which suggested that the glass powder was also extracted to the isooctane phase, and thus these two powders could not be well separated. On the other hand, the recovery of cerium oxide abrasive in the solid recovered from the isooctane phase is relatively low at pH 3–5, which implied that most of the cerium oxide abrasive stayed in the water phase. However, the content and recovery of cerium oxide abrasive in the solid recovered from the water phase at pH 3 were 81.5% and 76.3%, respectively, and those at pH 5 were 81.6% and 72.4%, respectively, in which the content was near its initial content and the recovery was relatively low. The results suggested that by using DAA as the surfactant collector, the cerium oxide abrasive and the polished glass could not be well separated.
Figure 6. The content and recovery of cerium oxide abrasive in the solid recovered from the isooctane phase as a function of pH with NaOL addition (Experimental conditions: abrasive-glass polishing waste ca. 2 g, DAA dosage 0.5 kg/ton, isooctane 20 mL, water 80 mL).

3.2.3. NaOL Addition

Figure 7 illustrates the content and recovery of cerium oxide abrasive in the solid recovered from the isooctane phase as a function of pH with NaOL addition. When adding 2 kg/ton NaOL, the content of cerium oxide abrasive in the solid recovered from the isooctane phase was above 80% throughout the experimental pH range from 3 to 11, which was higher than its initial content (80.5%) in the as-received polishing waste sample. Specifically, higher content (ca. 95%) was obtained at a pH of 7. On the other hand, higher recovery (near 80%) was also obtained at a pH of 7. Decreasing or increasing the pH from 7 reduced both the content and the recovery. The results suggested that the optimal pH for separating the cerium oxide abrasive and the polished glass with NaOL addition was at a pH of 7.

Figure 7. The content and recovery of cerium oxide abrasive in the solid recovered from the isooctane phase as a function of pH with NaOL addition (Experimental conditions: abrasive-glass polishing waste ca. 2 g, NaOL dosage 2 kg/ton, isooctane 20 mL, water 80 mL).

Figure 8 illustrates the content and recovery of cerium oxide abrasive in the solid recovered from the isooctane phase as a function of NaOL dosage at pH 7. When NaOL of 0.5 kg/ton was added, the content and recovery of cerium oxide abrasive was 81.6% and 69.3%, respectively. With the increase in NaOL dosage, the content and recovery of cerium oxide abrasive both increased. When NaOL of 7.5 kg/ton was added, optimal results were obtained; the content and recovery reached 96.4% and 88.1%, respectively, indicating that the cerium oxide abrasive and the polished glass were ideally separated.
The preceding results suggested that NaOL addition could improve the mutual separation of the cerium oxide abrasive and the polished glass in the abrasive-glass polishing waste, because NaOL could selectively improve the extraction of cerium oxide abrasive.

4. Discussion

In this section, the mechanism of each surfactant collector addition on improving the extraction from water to isooctane phase of the cerium oxide abrasive and the glass powder, as well as the mutual separation of these two powders, is discussed.

4.1. The Mechanism of Surfactant Collector Addition on Improving Extraction

According to the results shown in Section 3.1, DAA addition could improve the extracted fraction from water to isooctane phase of both cerium oxide abrasive and glass powder at pH 7–9 and pH 3–9, respectively, whereas NaOL addition could only improve that of cerium oxide abrasive across a broad pH range. A surfactant collector contains a hydrophilic group and a hydrophobic group. In general, a surfactant collector reacts with the particle through either electrostatic physical adsorption or chemical adsorption occurred between its hydrophilic group and the particle, and thus the surface of the particle becomes hydrophobic. By contrast, the hydrophobic group of the surfactant collector contacts with the air bubble or oil droplet, and thus the particle floats upwards. The occurrence of electrostatic physical adsorption and chemical adsorption is related to the surface charge of the particle and the dissociation status of the surfactant collector in aqueous solution. Usually, both of them vary with the solution pH.

Figure 9 illustrates the zeta potentials (i.e., the surface charges) of the cerium oxide abrasive and the glass powder, as measured by a zeta potential analyzer (Stabino, Particle Metrix GmbH, Meerbusch, Germany). Distilled water was used as the solvent. Potassium nitrate (KNO₃) solution of 1 × 10⁻³ M was used as the supporting electrolyte. The isoelectric point (IEP) of cerium oxide abrasive was observed at pH 6.2, and the surface of cerium oxide abrasive was determined to be negatively and positively charged above and below pH 6.2, respectively. By contrast, the IEP of the glass powder was observed at pH 2, and the surface of glass powder was determined to be negatively charged across a broad pH range above pH 2. The cerium oxide abrasive is produced from rare earth minerals such as monazite. The IEP and zeta potentials of the cerium oxide abrasive are similar to the reported analysis results of the monazite [20].
With respect to the surfactant collector of DAA, DAA (C\textsubscript{14}H\textsubscript{31}NO\textsubscript{2}) is substantially composed of two components: dodecylamine (C\textsubscript{12}H\textsubscript{25}NH\textsubscript{2}) and acetic acid (CH\textsubscript{3}COOH). Dodecylamine and acetate acid are weak base and weak acid, respectively, which have acid dissociation constants (pKa) of 10.63 and 4.8, respectively [19]. When DAA is in an aqueous solution, the following reactions occur

\[
\text{C}_{12}\text{H}_{25}\text{NH}_2 + \text{H}^+ = \text{C}_{12}\text{H}_{25}\text{NH}_3^+ \quad (1)
\]
\[
\text{CH}_3\text{COOH} = \text{CH}_3\text{COO}^- + \text{H}^+ \quad (2)
\]

The species of DAA in an aqueous depends on the solution pH. Between pH 4.8 and 10.63, the acetic acid dissociates to form the acetate ion (CH\textsubscript{3}COO\textsuperscript{−}) and releases a hydrogen ion (H\textsuperscript{+}). The dodecylamine accepts the hydrogen ion to form C\textsubscript{12}H\textsubscript{25}NH\textsubscript{3}\textsuperscript{+}. The reaction of DAA with particle is mainly through its hydrophilic group, i.e., the amine group NH\textsubscript{3}, and thus DAA acts as a cationic surfactant collector. Below pH 4.8, the acetic acid dissociates less, and thus the hydrogen ion is less released. The hydrophilic group of dodecylamine exists as NH\textsubscript{2}, and thus the capability of DAA as a cationic surfactant collector reduces. Above pH 10.63, the Equation (1) progresses inversely so that the hydrophilic group of dodecylamine exists as NH\textsubscript{2}, and thus the capability of DAA as a cationic surfactant collector also decreases.

Below pH 6, the surface of cerium oxide abrasive is positively charged. Meanwhile, the DAA acts as a cationic surfactant collector. Therefore, the electrostatic physical adsorption between the positively charged cerium oxide abrasive and the cationic surfactant collector DAA cannot occur. Hence, the extracted fraction of cerium oxide abrasive is not substantially improved. Above pH 6, the surface of cerium oxide abrasive becomes negatively charged, and the electrostatic physical adsorption with the cationic surfactant collector DAA is able to occur. Therefore, the extracted fraction of cerium oxide abrasive is significantly improved. The higher the pH, the higher extracted fraction of cerium oxide abrasive is obtained. Above pH 10, the capability of DAA as a cationic surfactant collector reduces, and thus the effectiveness in improving the extracted fraction of cerium oxide abrasive is decreased. By contrast, the surface of glass powder is negatively charged above pH 2. The electrostatic physical adsorption between glass powder and the cationic surfactant collector DAA is likely to occur, and thus the extracted fraction of glass powder is improved. However, below pH 4.8 and above pH 10.63, the capability of DAA as a cationic surfactant collector reduces, and thus the effectiveness in improving the extracted fraction of glass powder is decreased. Consequently, the effectiveness of DAA addition in improving the extracted fraction of the cerium oxide abrasive and the glass powder is attributed to the electrostatic physical adsorption between DAA and them.

With respect to the surfactant collector of NaOL, NaOL (C\textsubscript{17}H\textsubscript{33}COONa) is a strong electrolyte which fully ionizes to form the anionic oleic ion (C\textsubscript{17}H\textsubscript{33}COO\textsuperscript{−}) in an aqueous solution and thus acts as an anionic surfactant collector. Below a pH of 6, the electrostatic physical attraction between positively

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**Figure 9.** Zeta potentials of the cerium oxide abrasive and the glass powder.

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\[
\text{C}_{12}\text{H}_{25}\text{NH}_2 + \text{H}^+ = \text{C}_{12}\text{H}_{25}\text{NH}_3^+ \quad (1)
\]
\[
\text{CH}_3\text{COOH} = \text{CH}_3\text{COO}^- + \text{H}^+ \quad (2)
\]

The species of DAA in an aqueous depends on the solution pH. Between pH 4.8 and 10.63, the acetic acid dissociates to form the acetate ion (CH\textsubscript{3}COO\textsuperscript{−}) and releases a hydrogen ion (H\textsuperscript{+}). The dodecylamine accepts the hydrogen ion to form C\textsubscript{12}H\textsubscript{25}NH\textsubscript{3}\textsuperscript{+}. The reaction of DAA with particle is mainly through its hydrophilic group, i.e., the amine group NH\textsubscript{3}, and thus DAA acts as a cationic surfactant collector. Below pH 4.8, the acetic acid dissociates less, and thus the hydrogen ion is less released. The hydrophilic group of dodecylamine exists as NH\textsubscript{2}, and thus the capability of DAA as a cationic surfactant collector reduces. Above pH 10.63, the Equation (1) progresses inversely so that the hydrophilic group of dodecylamine exists as NH\textsubscript{2}, and thus the capability of DAA as a cationic surfactant collector also decreases.

Below pH 6, the surface of cerium oxide abrasive is positively charged. Meanwhile, the DAA acts as a cationic surfactant collector. Therefore, the electrostatic physical adsorption between the positively charged cerium oxide abrasive and the cationic surfactant collector DAA cannot occur. Hence, the extracted fraction of cerium oxide abrasive is not substantially improved. Above pH 6, the surface of cerium oxide abrasive becomes negatively charged, and the electrostatic physical adsorption with the cationic surfactant collector DAA is able to occur. Therefore, the extracted fraction of cerium oxide abrasive is significantly improved. The higher the pH, the higher extracted fraction of cerium oxide abrasive is obtained. Above pH 10, the capability of DAA as a cationic surfactant collector reduces, and thus the effectiveness in improving the extracted fraction of cerium oxide abrasive is decreased. By contrast, the surface of glass powder is negatively charged above pH 2. The electrostatic physical adsorption between glass powder and the cationic surfactant collector DAA is likely to occur, and thus the extracted fraction of glass powder is improved. However, below pH 4.8 and above pH 10.63, the capability of DAA as a cationic surfactant collector reduces, and thus the effectiveness in improving the extracted fraction of glass powder is decreased. Consequently, the effectiveness of DAA addition in improving the extracted fraction of the cerium oxide abrasive and the glass powder is attributed to the electrostatic physical adsorption between DAA and them.

With respect to the surfactant collector of NaOL, NaOL (C\textsubscript{17}H\textsubscript{33}COONa) is a strong electrolyte which fully ionizes to form the anionic oleic ion (C\textsubscript{17}H\textsubscript{33}COO\textsuperscript{−}) in an aqueous solution and thus acts as an anionic surfactant collector. Below a pH of 6, the electrostatic physical attraction between positively
charged cerium oxide abrasive and anionic oleic ion is likely to occur. Therefore, the extracted fraction of cerium oxide abrasive is improved. However, it is observed that the extracted fraction decreased with the decrease in solution pH below pH 6. It is considered that the oleic ion can react with hydrogen ion to form the oleic acid (C_{17}H_{33}COOH) according to the following Equation (3)

\[
C_{17}H_{33}COO^- + H^+ = C_{17}H_{33}COOH
\]

The pKa of oleic acid is 5 [21]. Therefore, in a weak acid medium near and below pH 5, the oleic ion starts to react with the hydrogen ion to form the oleic acid. Hence, the amount of oleic ion reduces, and thus its improvement of the extraction of the cerium oxide abrasive decreases. On the contrary, above a pH of 6, the cerium oxide abrasive becomes negatively charged and the electrostatic physical adsorption with the anionic oleic ion is not likely to occur. However, the extracted fraction of cerium oxide abrasive is still improved. It is considered that chemical adsorption between cerium oxide abrasive and oleic ion may occur. It was reported that oleic ion can react with metal (Me) to form Me(C_{17}H_{33}COO)_{2} [22]. In addition, oleic ion can also absorb to the surface of monazite chemically [23]. Hence, the extracted fraction of the cerium oxide abrasive can be improved with NaOL addition.

At higher pH values, the negative charge of cerium oxide abrasive increases. The electrostatic repulsion between cerium oxide abrasive and anionic oleic ion also increases; thus, the extracted fraction of cerium oxide abrasive decreases. Consequently, the effectiveness of NaOL addition in improving the extracted fraction of the cerium oxide abrasive is attributed to the synergistic effect of electrostatic physical adsorption and chemical adsorption between NaOL and cerium oxide abrasive. A higher extracted fraction of the cerium oxide abrasive is obtained at pH 7. By contrast, the glass powder cannot react with NaOL by means of either electrostatic attraction or chemical adsorption; thus, its extracted fraction cannot be improved with NaOL addition. Therefore, NaOL addition can selectively improve the extraction of the cerium oxide abrasive.

4.2. The Mechanism of Surfactant Collector Addition on Improving Separation

According to the results shown in Section 3.2, the cerium oxide abrasive and the polished glass in the abrasive-glass polishing waste could not be separated without surfactant collector addition. In addition, DAA addition could not improve their separation very well, whereas NaOL could with a specific pH range and dosage. The results are considered to be caused by the combined effects of the extraction of the cerium oxide abrasive and the glass powder, as well as the surface charge of these two powders.

When surfactant collector was not added, the recovery of cerium oxide abrasive in the solid recovered from the isooctane phase was ca. 10%, and the content of that was near its initial content (80.5%) in the polishing waste. This is because neither cerium oxide abrasive nor glass powder can be extracted to the isooctane phase without a surfactant collector addition. Most of these two powders remain in the water phase and therefore cannot be separated.

When using DAA as the surfactant collector, the extraction of the cerium oxide abrasive could be improved at pH 7–9, and that of the glass powder could be improved at pH 3–9. When the separation was conducted at pH 7–9, although a higher recovery (more than 90%) of cerium oxide abrasive in the solid recovered from the isooctane phase can be obtained, the content was near its initial content in the polishing waste. This is because both of these two powders were extracted from water to the isooctane phase at pH 7–9 simultaneously, which means the mutual separation of these two powders cannot be achieved. On the other hand, when the separation was conducted at pH 3–5, where most of the polished glass was significantly extracted to the isooctane phase whereas most of cerium oxide abrasive was not, the cerium oxide abrasive might be recovered from the water phase. However, the content of cerium oxide abrasive in the solid recovered from the water phase at pH 3–5 was near its initial content and its recovery was relatively low, which suggested that the ideal separation also
cannot be achieved. It is considered that the results are caused by the heteroaggregation between the cerium oxide abrasive and glass powder.

According to the zeta potentials shown in Figure 9, the surface charge levels of the cerium oxide abrasive and the glass powder were positive and negative, respectively, below a pH of 6.2. Consequently, the cerium oxide abrasive aggregated with the glass powder below pH 6.2, owing to electrostatic attraction. This might result in two outcomes. The first one is that the cerium oxide abrasive is extracted to the isooctane phase with the glass powder, leading to a decrease in the recovery of cerium oxide abrasive in the solid recovered from the water phase. The second one is that the extraction of the glass powder is hindered by the cerium oxide abrasive, resulting in a decrease in the content of cerium oxide abrasive in the solid recovered from the water phase. According to the experimental results, the content and recovery of cerium oxide abrasive in the solid recovered from the water phase was near its initial content and its recovery was relatively low at pH 3–5, implying that both outcomes occurred simultaneously. Therefore, these two powders cannot be well separated.

When using NaOL as the surfactant collector, only the extraction of the cerium oxide abrasive could be improved with NaOL addition across a wide pH range, whereas that of glass powder could not. Below the pH of 6.2, the content and recovery of cerium oxide abrasive in the solid recovered from the isooctane phase both decreased. It is considered that the aforementioned two outcomes of heteroaggregation, i.e., the cerium oxide abrasive extracted to the isooctane phase with the glass powder, leading to a decrease in the content of cerium oxide abrasive in the solid recovered from the isooctane phase, as well as the extraction of the cerium oxide abrasive being hindered by the glass powder, resulting in a decrease in the recovery of cerium oxide abrasive in the solid recovered from the isooctane phase, occurred simultaneously. Above a pH of 6.2, the heteroaggregation between the two powders became weak, and only the cerium oxide abrasive was extracted to the isooctane phase; therefore, a higher content and recovery of cerium oxide abrasive in the solid recovered from the isooctane phase was obtained. Above a pH of 7, the cerium oxide abrasive showed lower content and recovery owing to the decrease in its extracted fraction. Therefore, the ideal mutual separation of cerium oxide abrasive and polished glass is accomplished at a pH of 7.

4.3. The Strategy for Recovering Cerium Oxide Abrasive from Abrasive-Glass Polishing Waste

Concerning the mutual separation of cerium oxide abrasive and glass powder in an abrasive-glass polishing powder waste for recovering cerium oxide abrasive, there are two approaches to achieve this goal, applying the liquid–liquid–powder extraction method. The first one is to extract the glass powder to the isooctane phase and leave the cerium oxide abrasive in the water phase. The second one is to extract the cerium oxide abrasive to the isooctane phase and leave the glass powder in the water phase. Actually, an abrasive-glass polishing waste is generated from applying the cerium oxide abrasive to polish the glass component. The polishing ability of cerium oxide abrasive diminishes with the increase in polished glass. In general, the amount of polished glass should be less than that of the cerium oxide abrasive, otherwise the quality of polishing product cannot be ensured. Therefore, the content of the cerium oxide abrasive in a polishing waste is often more than that of the polished glass. Similar to the concept in the froth flotation method, in which the smaller component is floated to the froth, the recovery of cerium oxide abrasive from abrasive-glass polishing waste using the liquid–liquid–powder extraction method can ideally be carried out through extracting the lesser component, i.e., the polished glass, to the isooctane phase and leaving the cerium oxide abrasive in the water phase. Therefore, the aforementioned first approach is preferred, i.e., using a cationic surfactant collector of DAA and conducting the separation at pH 3–5 to selectively improve the extraction of polished glass. The improvement by DAA addition is based on physical electrostatic adsorption with the polished glass. However, the heteroaggregation between cerium oxide abrasive and polished glass occurs at pH 3–5, which leads to the extraction of the cerium oxide abrasive to the isooctane phase with the polished glass, and thus these two powders cannot be well separated. Consequently, the optimal approach for the recovery of cerium oxide abrasive is through the aforementioned second
approach, i.e., using an anionic surfactant collector of NaOL and conducting the separation at pH 7 to selectively improve the extraction of cerium oxide abrasive. The improvement by NaOL addition is based on chemical adsorption with the cerium oxide abrasive. In addition, the heteroaggregation between cerium oxide abrasive and polished glass does not occur at pH 7. Only the cerium oxide abrasive is extracted to the isooctane phase, whereas the polished glass remains in the water phase, and therefore good separation and recovery of the cerium oxide abrasive can be accomplished.

5. Conclusions

This study applied the liquid–liquid–powder extraction method to separate the cerium oxide abrasive and the polished glass powder in an abrasive-glass polishing waste to recover the cerium oxide abrasive. Two liquids of isooctane and water were utilized. The effectiveness of using a cationic and an anionic surfactant collector, i.e., dodecylamine acetate (DAA) and sodium oleate (NaOL), respectively, on improving their extraction and separation was investigated and compared. The following results were obtained:

(1) The extracted fraction from water to isooctane phase of the cerium oxide abrasive could be improved from ca. 10% to nearly reach 100% by adding DAA of 0.5 kg/ton at pH 7–9, and to nearly reach 100% by adding NaOL of 2 kg/ton at pH 7, whereas that of the glass powder could only be improved from ca. 10% to nearly reach 100% by adding DAA of 0.5 kg/ton at pH 5–9;

(2) The addition of NaOL could improve the mutual separation of the cerium oxide abrasive and the glass powder, whereas that of DAA could not. Optimal separation for the cerium oxide abrasive and the polished glass powder in an abrasive-glass polishing waste was achieved by adding NaOL of 7.5 kg/ton at pH 7; the content of cerium oxide abrasive in the solid recovered from the isooctane phase was 96.4% with a recovery of 88.1%.

The experimental results suggest that adding an anionic surfactant collector, NaOL, can selectively increase the extraction of the cerium oxide abrasive from the water to isooctane phase and thus improve the separation of the cerium oxide abrasive and the polished glass powder in an abrasive-glass polishing waste.

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