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Recovery of polyalkylene glycol from silicon cutting waste using centrifugation

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ABSTRACT

A great amount of slurry waste is formed during the cutting process from silicon ingot to wafers. More than 50% of this waste comes from cutting liquids. This study investigated centrifugation to recover polyalkylene glycol (PAG) cutting liquid with low turbidity (<100 NTU). The experimental results show that clean PAG liquids could be obtained by using water as a diluent. Because water with high chemical polarity and strong hydrogen bonding would destroy the adsorption of PAG molecules on particles, and weaken the steric stabilization, the particles are aggregated and then separated from liquids by centrifugation. After 50 wt.% water-assisted centrifugation at 3,253 G-force for 24 h, the solid content of the upper liquid decreased to 0.018 g/L, and the turbidity reduced to 7.2 nephelometric turbidity units (NTU). The obtained liquid was then vacuum distillated to remove water. The final recovered PAG with only 0.43 NTU could be reused in the cutting process.

Keywords: Recovery; Polyalkylene glycol; Cutting liquid; Silicon cutting waste; Centrifugation

1. Introduction

Due to the fast growth rate of the photovoltaic industries, the need for silicon wafers increased. In silicon wafer manufacturing, a polycrystalline silicon ingot is cut by a multi-wire saw with slurry containing glycol cutting liquids and silicon carbide (SiC) abrasives. During this cutting operation, kerf-loss silicon (Si) and metal fragments from the cutting wire are incorporated into the slurry, resulting in a large amount of waste [1]. This silicon cutting waste is usually disposed off by incineration or treated by a wastewater treatment facility, causing a non-negligible environmental impact [2].

In recent years, several researchers devoted on the separation of Si and SiC to recover Si or SiC [3–5], but the recovery of cutting liquids in these studies is not included. In fact, more than 50% of this waste comes from cutting liquids. Regeneration and recovery of cutting fluids via simple and cheap processes should allow the reuse of used fluid, and might substantially reduce the amount of silicon cutting waste and photovoltaic industry wastewater [6].

The cutting liquid generally consists of water-soluble glycol molecules, which can be classified as small molecules or polymers. Polyalkylene glycol (PAG) belongs to a polymer-type cutting liquid and is

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most often used for silicon slicing. This PAG cutting liquid possesses long carbon chains in its molecule, resulting in a good dispersion of abrasives, high lubricity, and heat tolerance, thereby exhibiting an excellent cutting yield [7]. However, the used PAG liquid forms wastewater with high chemical oxygen demand, which is difficult to treat and becomes a considerable environmental issue [8,9]. Recycling this glycol liquid from cutting waste would greatly reduce the amount of waste, and thus the total slicing cost can be efficiently reduced.

Many manufacturers and recyclers use distillation to recover small-molecule cutting liquids, the recovery of diethylene glycol (DEG), for example. During distillation, the cutting waste is heated to vaporize DEG, and then the obtained vapor can be separated from the solid waste. By condensation, high-purity DEG can be collected [10]. However, polymer-type cutting liquid dissociates or oxidizes at high temperature and cannot be recovered through the collected vapor during distillation. Therefore, some manufacturers and recyclers use low-temperature methods to separate cutting liquids from silicon cutting waste such as centrifugation [11,12] and filtration [9,12]. Recently, Drouiche et al. used ultrafiltration reinforced by a chemical pretreatment to regenerate cutting fluids, and their recovered liquid showed similar infrared spectra to pure cutting fluids [13]. Still, many separation methods of solid and liquid are suggested to recover a PAG liquid, but it is difficult to recover a clean PAG liquid due to the stable suspension of SiC, Si particles, and metal fragments in PAG. Over the past few years, patents proposed various techniques or skills to recover and renew polymer cutting liquids, including predilution [9,11,12] for enhancing separation of solid and liquid, coagulation [14] or membrane filtration [15] for separation of particles, ion exchange treatment [16], reverse osmosis treatment [15] or vacuum evaporation [17] for removing water, and decolorizing for adsorbing impurities. The suggested methods were only patented, and no practical data could be provided for the recovery of PAG liquids from silicon cutting waste.

This study adopted centrifugation treatment to recover PAG cutting liquids from silicon cutting waste. Centrifugal separation provides several advantages to separate solid and liquid, such as simple operation and rapid separation, and is easily performed in the industry. To obtain a low-turbidity PAG liquid, less than 100 nephelometric turbidity units (NTU), from silicon cutting waste, this study adjusted the operational variables of centrifugation and employed acetone, ethanol, and deionized water as diluents for enhancing separation performance. Observation of the aggregation behaviors revealed the separation mechanism of particles during PAG recovery from silicon cutting waste. Finally, a clean recovered liquid was obtained for use as the new PAG.

2. Experimental

2.1. Analysis of silicon cutting waste

Silicon cutting waste was obtained from Sino-American Silicon Products, Inc. (Chu-Nan, Taiwan). After preliminary recycling of large SiC particles with a centrifugal decanter, the slurry waste with low solid content was transferred to our laboratory for the sequential analysis and recovery of cutting liquids.

The obtained silicon cutting waste contained kerf Si, SiC abrasives, metal fragments from cutting wire, and PAG cutting liquid. To analyze its composition, each waste sample was first heated to 500℃ to remove liquids. The solid content percentage (S_1) was then calculated as the mass of the residual solid powder dividing by the mass of the original obtained waste. The particle size distribution (PSD) in the obtained silicon cutting waste was measured using static light scattering (model: LA300, HORIBA). Viscosity was measured using a viscometer (model: LVDV-I Prime, BROOKFIELD), and turbidity was measured with a turbidity meter (model: 2100Q, HACH).

2.2. Centrifugal separation

Silicon cutting waste (30 mL) was placed in a centrifuge tube to be transferred to a centrifuge. Herein, Hermle Z323K centrifuge with 25° fixed angle rotor (maximum radius, $r_{\max}$, 96 mm), operated at 5,500 rpm, is applied for the separation of solid and PAG liquid. The corresponding structure of the centrifuge was shown in Fig. 1.

Assume, the particles settle in the region of Stokes law and move along the axial direction of centrifuge tube. The particle settling velocity in a centrifugal field could be estimated by the following equation [18].

$$V_t = \frac{\mathrm{d}x}{\mathrm{d}t} = \frac{(\rho_p - \rho_l) \times \mathrm{d}_p{}^2 \times r\omega^2}{18\mu_l}(1-\varphi)^{4.65} \tag{1}$$

where d_p, ρ_p, ρ_l, and μ_l are particle diameter, particle density, liquid density, and liquid viscosity, respectively. In addition, $(1-\varphi)^{4.65}$ is the hindered settling function developed by Richardson and Zaki [19], and φ is the solid volume fraction in suspension. The radius, r, is equal to $x \sin\theta$, L is the length occupied by the waste sample in the centrifuge tube, and T will be

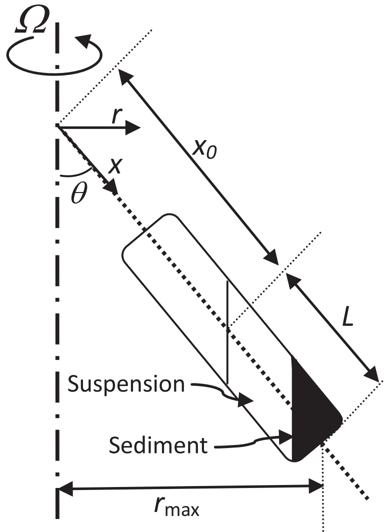

Fig. 1. Schematic diagram of the centrifuge.

the time for the particle to travel through the tube from the top ($x = x_0$)to the bottom ($x = x_0 + L$). After integrating between the two boundary conditions, $x = x_0$ at $t = 0$ and $x = x_0 + L$ at $t = T$, T can be expressed as the following equation:

$$T = \frac{18\mu_l}{\left(\rho_p - \rho_l\right) \times {d_p}^2 \times \omega^2 \sin\theta}(1-\varphi)^{-4.65} \ln\left[\frac{x_0 + L}{x_0}\right] \tag{2}$$

According to Eq. (2), the effects of centrifugation time (T) and the solid volume fraction (φ), controlled by number of centrifugation steps on the solid content of the recovered liquid, were investigated. Moreover, to adjust the liquid density and viscosity, three liquids—acetone, ethanol, and deionized water—were used as diluents and mixed with the obtained silicon cutting waste by 5 min stirring. After thoroughly mixing the waste and diluents, 30-mL diluted waste was put in a tube for the centrifugation experiments.

2.3. Analysis of recovered liquids

After centrifugation for various durations, the upper liquid or the recovered liquid in the centrifuge tube was analyzed. PSD was measured by static light scattering (model: LA300, HORIBA). Viscosity was measured with a viscometer (model: LVDV-I Prime, BROOKFIELD). Turbidity was measured with a turbidity meter (model: 2100Q, HACH). In addition, the recovery liquid was heated to 500˚C to remove glycol or diluents, and the solid content percentage (S_2) was obtained by the mass of the residual solid divided by the mass of the original recovered liquid.

Due to the hygroscopicity of PAG molecules, the obtained waste or recovered liquid would contain water, and the water content was measured with a Karl Fischer titrator (model: 870 plus, HACH). When deionized water was adopted as the diluent, the following equation (Eq. (3)) was used to calculate the recovery percentage of glycol (Y) based on water content percentage in the obtained waste (W_1) and in the recovered liquid (W_2).

$$Y\,(\%) = \frac{M_r \times (1 - S_2 - W_2)}{M_o \times (1 - S_1 - W_1)} \times 100\% \tag{3}$$

where M_o is the mass of the obtained waste and M_r is the mass of the recovered liquid.

3. Results and discussion

The obtained cutting waste shown in Fig. 2(a) had a black and opaque appearance. The measured density, viscosity at 25˚C, and solid content of the

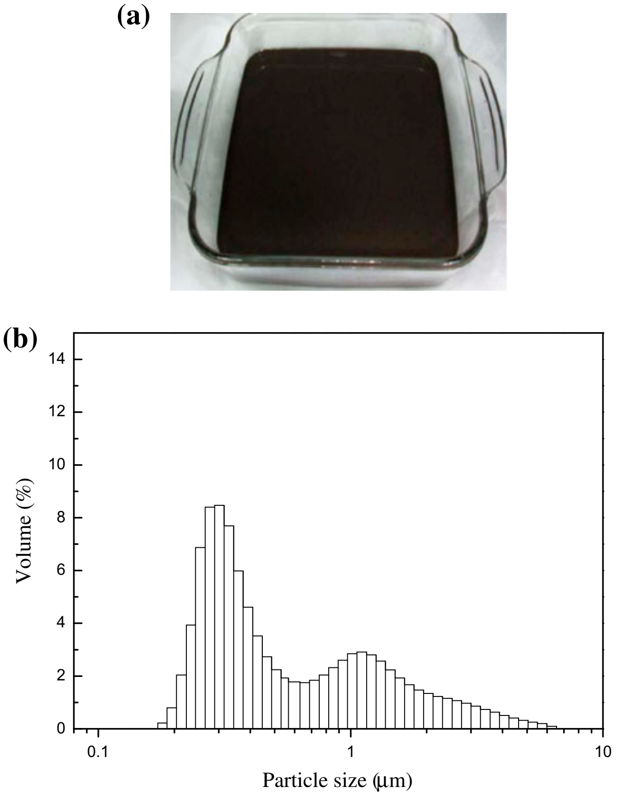

Fig. 2. The obtained slurry waste: (a) the appearance and (b) PSD.

waste were 1.009 g/cm^3, 72 cp and 15.6 wt.%, respectively. The PSD of the waste (Fig. 2(b)) indicated that the particle size of the obtained waste was below 7.0 μm, and the main peaks of the particle sizes were 0.3 and 1.2 μm, which showed that most of the large SiC (around 15 μm as used originally) was recycled before transferring to our laboratory. These micron particles in spent cutting waste included kerf Si, metal fragments, and broken SiC abrasives. Because of their small size, the particles were suspended stably in PAG cutting liquid, which produced an opaque appearance and high NTU. The turbidity of the obtained waste exceeded 1,000 NTU, which was the detectable maximum of the turbidity meter (model: 2100Q, HACH).

To recover a clean PAG liquid, lowering the solid content and turbidity is essential. Herein, a standard value corresponding to a clean PAG liquid was examined by adding approximately 1 μm silicon particles into PGA to form a PAG–Si suspension. The prepared PAG–Si suspension was used in analyzing the relations among solid content, turbidity, and appearance of PAG–Si suspension. Fig. 3 displays the effects of solid contents and turbidity on the appearance of the prepared PAG–Si suspension. As shown, the appearance of the suspension was not transparent, and the turbidity exceeded 1,000 NTU when the solid content reached 0.5 g/L (Fig. 3(e)). The appearance was clean and resembled an unused cutting liquid when the solid content decreased to 0.0075 g/L, and the turbidity reduced to 109 NTU (Fig. 3(c)). The smaller particles at the same solid content exhibited a higher turbidity. Since the waste analyzed in this study consisted mainly of 0.3 and 1.2 μm particles, the turbidity requirement of the recovered liquids was set below 100 NTU.

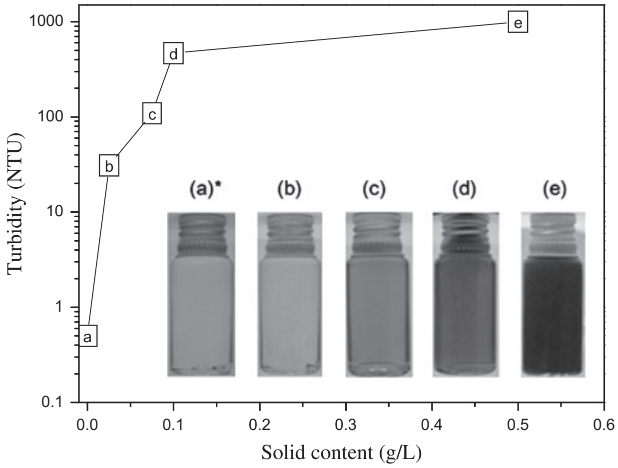

Fig. 3. The effect of the solid content on turbidity and appearance of the prepared PAG-Si suspensions. (a)–(e) Photos corresponding to the letters marked on the curve. (*Unused PAG liquid).

According to Eq. (2), during centrifugation at 5,500 rpm, 0.3 and 1.2 μm Si particles, starting at the top of centrifuge tube, were estimated requiring 4.87 and 0.30 h to settle, respectively, for $\mu_l = 43$ cp, $L = 55$ mm, $r_{\max} = 96$ mm, $\theta = 25^\circ$ and $\varphi = 6.44\%$. The experimental results about the influence of centrifugation time were displayed in Fig. 4, showing that in 3 h, the solid content of the upper liquid decreased significantly after centrifugation. As estimated by Eq. (2), most 0.3 or 1.2 μm Si particles could settle by centrifugation in 4.87 h. When the centrifugation was performed longer than 6–24 h, the solid content of the upper liquid decreased only by 0.91 g/L. Finally, the solid content after 24 h centrifugation reduced to 1.439 g/L. The particle removal fraction reached 99.04%, but the turbidity of the collected upper liquid was still higher than 1,000 NTU. This result might be attributed by the fact that smaller particles (<0.3 μm) hardly settle down.

After centrifugation for various durations, PSDs of the collected upper liquids in Fig. 5 showed that most 1.2 μm particles were removed from liquids after 1 h, and the main peak of the particle size approximated to 0.26 μm after centrifugation for 6 h. These submicron-sized particles suspended stably and were difficult to separate from PAG liquid by centrifugation at 5,500 rpm (3253 G-force). The turbidity of the recovered liquid still exceeded 1,000 NTU.

Fig. 6 shows the effect of the number of centrifugation steps on the solid content of the upper liquid. After the first 1-h centrifugation, the solid content of the upper liquid decreased, obviously, from 150 to 13.3 g/L. The upper liquid was collected and centrifuged again to obtain the liquid with lower solid

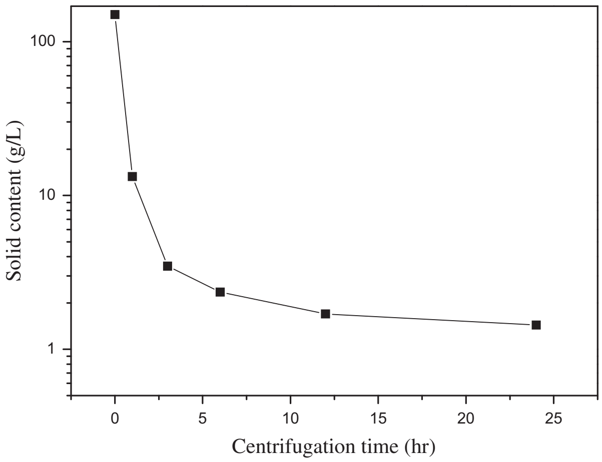

Fig. 4. The solid content of the recovered liquid after centrifugation for various durations.

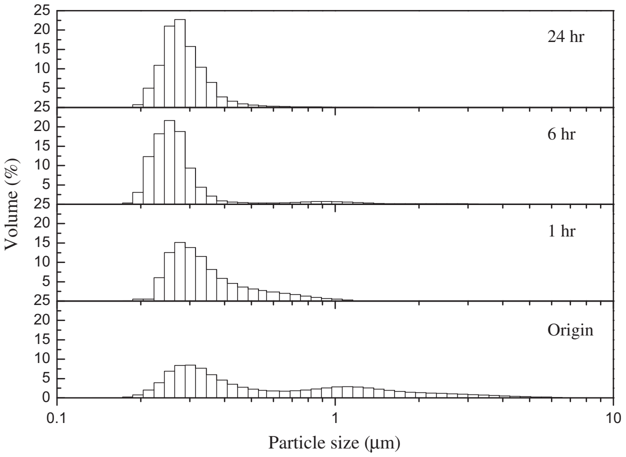

Fig. 5. PSD of original waste and the recovered liquid after centrifugation for various durations.

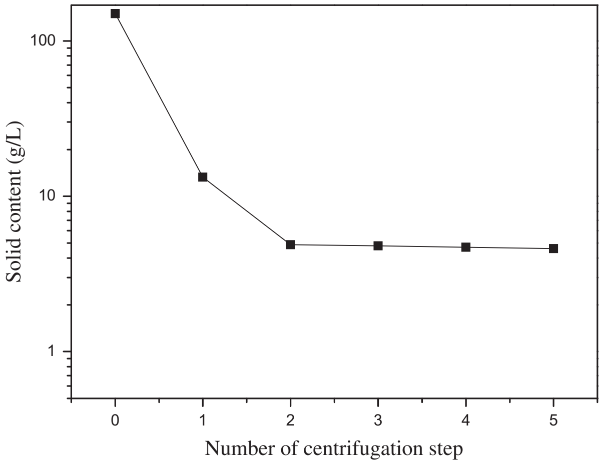

Fig. 6. The effect of the number of centrifugation step on the solid content of the upper liquid.

content. However, when the centrifugation step was repeated once, the decreasing extent of solid content of the collected liquid reduced. According to Eq. (2) with the centrifugation operation at 5,500 rpm, 0.3, and 1.2 μm Si particles, starting at the top of centrifuge tube with the initial solid content 150 g/L, were estimated to settle in 4.53 and 0.28 h, respectively. Therefore, after the first 1-h centrifugation, most 1.2 μm Si particles could settle to bottom, but 0.3 μm particles could not. When the number of centrifugation steps was 2, 0.3 μm Si particles still required 3.42 h to settle according to the calculation by Eq. (2). Therefore, around 1.2 μm Si particles were removed gradually, and the solid content decreased readily with the increasing number of centrifugation steps. Until the number of centrifugation step exceeded two, the particles in the collected upper liquid were too small to settle, causing that the decreasing extent of solid content in the upper liquid was very small. Even though the solid content after the 5th centrifugation reduced to 4.603 g/L, and the particle removal fraction reached 96.93%, the turbidity of the collected upper liquid still exceeded 1,000 NTU due the existence of submicron-sized particles.

The effect of the number of centrifugation steps on PSDs of the collected upper liquids (Fig. 7) shows that most 1.2 μm particles were removed from liquids after the first 1-h centrifugation, and the main peak of the particle size approximated to 0.29 μm after the 2$^{\text{nd}}$, and even after the 5th centrifugation. These submicron-sized particles suspended stably and were difficult to separate from PAG liquid by centrifugation at 5,500 rpm (3253 G-force). Therefore, the turbidity of the recovered liquid still exceeded 1,000 NTU.

Water, ethanol, and acetone were then used as diluents to decrease the viscosity and density of the slurry waste. Fig. 8 shows that the liquid density did not change significantly, while the liquid viscosity decreased with the addition of the diluents, especially when adding ethanol or acetone. Based on this finding, the average settling velocity ($\overline{V}_d$) was calculated in a centrifugal field at various concentrations of diluents using Eq. (2) and the following equation.

$$\overline{V}_d = L/T \tag{4}$$

where L is the length occupied by the waste sample in the centrifuge tube and T is the time for the particle to travel through the tube from the top $(x = x_0)$ to the

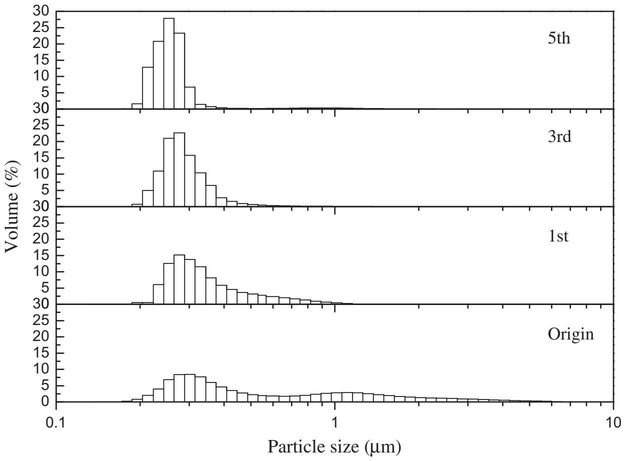

Fig. 7. The effect of the number of centrifugation step on PSDs of the collected upper liquids.

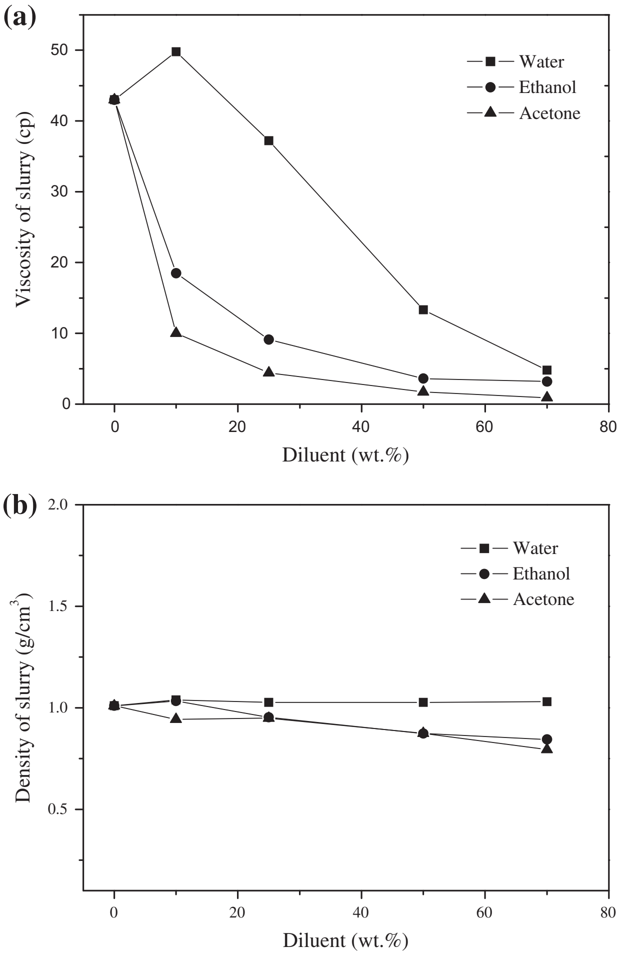

Fig. 8. The effect of adding diluents on (a) viscosity; (b) density of the slurry waste.

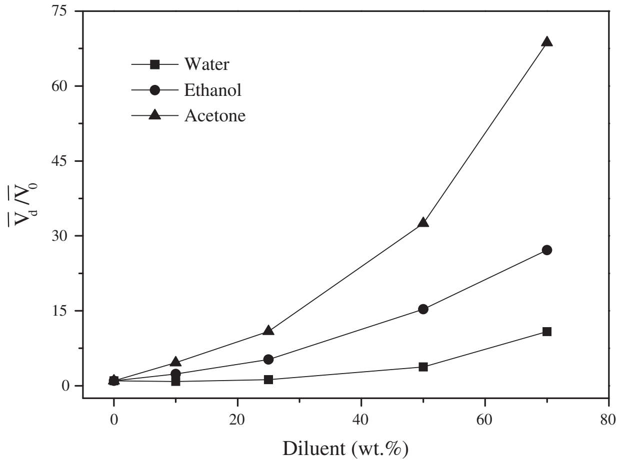

Fig. 9. The average settling velocity with respect to the original average velocity ($\overline{V}_d/\overline{V}_o$) at various concentrations of diluents.

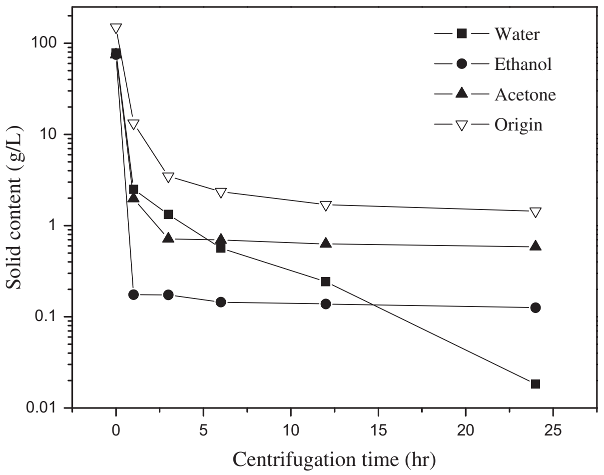

Fig. 10. The solid content of the upper liquid after centrifugation with 50 wt.% two diluent for various durations.

bottom ($x = x_0 + L$). Fig. 9 shows the effect of adding diluents on $\overline{V}_d$ with respect to the original average velocity ($\overline{V}_o$). The curves in Fig. 9 predicted that the addition of diluents could certainly increase the settling velocity in a centrifugal field, and adding acetone provided the fastest separation of solid and liquid. For example, when the diluent concentration was 50 wt.%, for 1.2 μm Si particles, $\overline{V}_d/\overline{V}_o$ was 32.50 for adding acetone and 15.31 for adding ethanol, but only 3.74 for adding water, which forecasted that recovery rate could be increased using a diluent, acetone especially.

Fig. 10 shows the effect of adding 50 wt.% diluent on the solid content of the upper liquid after centrifugation for various durations. The data indicated that all solid content decreased after the addition of diluents. In 3 h, the solid content decreased less for adding water among these three diluents. However, not as forecasted by $\overline{V}_d/\overline{V}_o$ in Fig. 9, the solid content was lowest by adding ethanol, not by adding acetone, in 3 h. After 1 h, the solid content was decreased to 0.2 g/L by adding ethanol and 1.97 g/L by adding acetone, but their decreasing extent reduced. After centrifugation for 24 h, the solid content reached 0.126 g/L by adding ethanol and 0.586 g/L by adding acetone. Despite the low solid content, the turbidity was still larger than 100 NTU with the addition of ethanol or acetone as displayed in Table 1. However, addition of water initially obtained a smaller decrease in solid content, but the effective reduction rate of solid content was sustained for 24 h. After 24 h, the solid content after addition of water was lower than that after addition of ethanol or acetone. After centrifugation for 24 h, the solid content reduced to 0.018 g/L by

Table 1
Solid content, turbidity and appearance of the unused PAG and the upper liquid after centrifugation with 50 wt.% diluents

		Upper liquid		
Liquid	Unused PAG	Water	Ethanol	Acetone
Solid content (g/L)	0	0.018	0.126	0.586
Turbidity (NTU)	0.5	7.2	762	>1,000
Appearance				

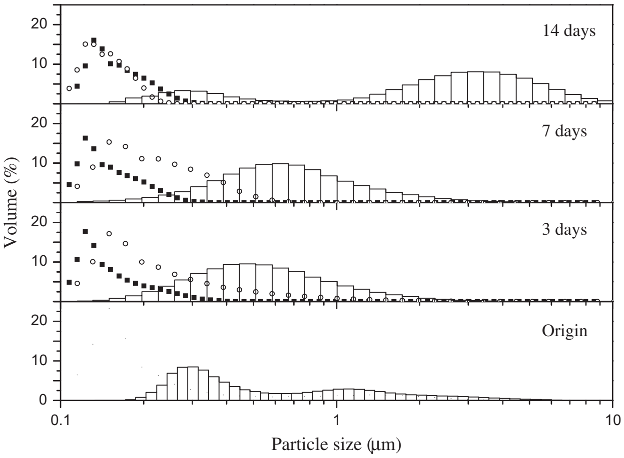

Fig. 11. PSD of the original waste and the waste mixed with 50 wt.% water (histogram), ethanol (scatter data: ■) or acetone (scatter data: ○) for various durations.

adding water, and the turbidity reduced to 7.2 NTU. Table 1 shows that the upper liquid with 7.2 NTU had a clean appearance and resembled an unused PAG liquid.

The above experimental results show that the mixture of waste and water provided a good separation performance due to the fast settling velocity of particles in a centrifugal field. To study the aggregation behaviors of particles during the waste contact with diluents, the mixtures of waste and different diluents were prepared to analyze PSDs.

The PSDs of the upper liquids after the waste mixed with 50 wt.% diluent for various durations are shown in Fig. 11. The data show that with increasing contact time, the main peak of the particle size was almost around 0.13 μm by adding ethanol or acetone. These submicron-sized particles suspended stably in PAG–ethanol or PAG–acetone solutions and were difficult to separate from PAG liquid by centrifugation at 5,500 rpm. However, with the addition of water, most particles in the upper liquid were 3.4 μm (Fig. 11), showing that the particles aggregated in PAG–water solution. In PAG–ethanol or PAG–acetone solutions, PAG molecules adsorbed on Si or SiC surfaces would prevent particle aggregation due to steric stabilization [20]. However, in PAG–water solution, formation of oxide enhanced the hydrophilicity of particles [21]. In addition, among these three diluents, water owns the largest chemistry polarity and hydrogen bonding component of Hansen solubility parameters [22], indicating different interactions using water as a diluent. In PAG–water solution, the attraction of hydrogen bonding happened between water and water or between PAG and water destroyed the adsorption of PAG molecules on particles, and then weakened the steric stabilization. The particles approached to each other in PAG–water solution easily and aggregated to form larger particles. The large particles readily settled in a centrifugal field, followed by separating from PAG to

Table 2
Properties of the unused and the recovered PAG by centrifugation and vacuum-distillation

Liquid (25°C)	Unused PAG	Recovered PAG
Viscosity (cp)	36.6	37.9
Density (g/cm^3)	0.994	0.997
Turbidity (NTU)	0.5	0.43
Water (wt.%)	0.251	0.186
Appearance		

water solution. Finally, a low-turbidity liquid was obtained.

After centrifugation with the addition of 50 wt.% water at 5,500 rpm for 24 h, the obtained upper liquid was vacuum-distillated at 60 torr and 70°C to remove water. The final recovered product, shown in Table 2, contained only 0.186 wt.% water. Table 2 indicates that the viscosity and density of the recovered liquid and the unused PAG were similar. Moreover, the turbidity of the recovered PAG was only 0.43 NTU. The estimated recovery percentage of glycol reached 95% according to Eq. (3).

During our process, water was the suggested diluent to mix with the spent cutting waste for the subsequent centrifugation separation. After centrifugation, water removal was performed by vacuum distillation at a low temperature such as 70°C or below 70°C. Thus, the obtained PAG liquid had a low turbidity and could be reused in wide applications.

4. Conclusion

This study recovered PAG cutting liquids from silicon cutting waste by centrifugation treatment. The separation performance of solid and liquid was enhanced by adjusting the operational variables of centrifugation and using diluents. Even though the particle removal fraction exceeded 99% by centrifugation or by adding ethanol or acetone, the recovered liquids were still opaque and turbid with larger than 100 NTU due to the stable suspension of submicron particles. The PSD results showed that the particles tended to aggregate in PAG–water solution. In PAG–ethanol or PAG–acetone solutions, PAG molecules adsorbed on Si or SiC surfaces prevent particle aggregation due to steric stabilization. In PAG–water solution, Si or SiC particles had hydrophilic surfaces, and the attraction of hydrogen bonding between water and water or between PAG and water would destroy the adsorption of PAG molecules on particles and weaken the steric stabilization. Thus, the particles would aggregate readily, causing rapid settling and facile separation from PAG to water solution. After centrifugation by adding 50 wt.% water for 24 h, the solid content of upper liquid decreased to 0.018 g/L, and the turbidity reached 7.2 NTU. The obtained upper liquid was then vacuum-distillated at 60 torr and 70°C to remove water. The final recovered PAG with 0.186 wt.% water was only 0.43 NTU. Meanwhile, the recovery percentage of glycol attained 95%.

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