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Author(s)	Ito, Mayumi; Saito, Ayumu; Takeuchi, Megumi; Murase, Nana; Phengsaart, Theerayut; Tabelin, Carlito Baltazar; Hiroyoshi, Naoki
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1 **Development of the reverse hybrid jig: Separation of polyethylene and cross-linked**

2 **polyethylene from eco-cable wire**

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4 Mayumi Ito [1,*], Ayumu Saito [2], Megumi Takeuchi [2], Nana Murase [2],

5 Theerayut Phengsaart [2,3], Carlito Baltazar Tabelin [1,4] and Naoki Hiroyoshi [1]

6

7 [1] Division of Sustainable Resources Engineering, Faculty of Engineering, Hokkaido

8 University, Kita 13, Nishi 8, Kita-ku, Sapporo 060-8628, Japan

9 [2] Division of Sustainable Resources Engineering, Graduate School of Engineering,

10 Hokkaido University, Kita 13, Nishi 8, Kita-ku, Sapporo 060-8628, Japan

11 [3] Department of Mining and Petroleum Engineering, Faculty of Engineering,

12 Chulalongkorn University, Bangkok 10330, Thailand

13 [4] School of Minerals and Energy Resources Engineering, The University of New South

14 Wales, Sydney, NSW 2052, Australia

15 * Corresponding author e-mail: itomayu@eng.hokudai.ac.jp

16

Abstract

We have developed various types of jigs for resources recycling focusing primarily on plastic-plastic separation. The RETAC jig could be used to separate plastics heavier than water (specific gravity (SG) > 1.0) with different SGs while the hybrid jig—a process combining jig and flotation—was developed to separate plastics with similar SGs but different surface wettabilities. Meanwhile the reverse jig—a RETAC jig with a top screen—is used to separate plastics lighter than water with different SGs.

In this study, a novel method that combines the principle of reverse and hybrid jig, called "reverse hybrid jig separation" was developed to separate plastics lighter than water having similar SGs but different surface wettabilities. The tests were carried out with wastes from an eco-cable wire recycling facility in Japan, which is composed of polyethylene (PE) and cross-linked polyethylene (XLPE). The reverse hybrid jig separation results showed that bubble attachment to and detachment from plastic particles could affect separation efficiency. In addition, the effects of particle size on separation efficiency were also evaluated.

1. Introduction

Plastics have become ubiquitous in our daily life with applications ranging from simple packaging materials to more complicated applications in electronic devices and automobiles (Jeon et al., 2018a; Tabelin et al., 2021a; Thiounn and Smith, 2020). The total production and consumption of plastics, including waste generation, have increased over the last couple of decades (Plastic waste partnership working group, 2020).

Fossil fuel-based plastics are further classified into either thermoplastic or thermosets. Thermoplastics are polymers that soften when heated and solidify upon cooling, a property that allows them to be remolded and recycled (i.e., material recycling). In contrast, thermosets are polymers that set into a mold once and cannot be re-softened or remolded again (Plastic waste partnership working group, 2020).

There are two common types of plastic recycling: (1) material recycling wherein plastics are recovered and reused, and (2) thermal recycling whereby plastics are used as fuel for power generation. Thermal recycling is more widely used to manage plastic wastes than material recycling. However, some plastics like polyvinyl chloride (PVC), are unsuitable for thermal treatment because they could generate harmful compounds like chlorine gas (Cl_2) and dioxins that have adverse effects to industrial processes, the environment and human health, so they usually end up in landfills (Ito et al., 2019a;

53   Tabelin et al., 2021b; Phengsaart et al, 2018).

54   Aside from challenges in recycling PVCs, this plastic also contains tribase—a

55   lead compound added into PVC as a thermal stabilizer (Tsunekawa et al., 2011). Lead is

56   a toxic heavy metal notorious for causing irreversible damage to the still-developing

57   nervous systems of fetus, babies and children (Silwamba et al., 2020a, b; Tabelin et al.,

58   2018, 2020). Because of this, the use of PVC as sheath and insulation of electric wires

59   has been gradually replaced by more eco-friendly plastics and are marketed as "eco-

60   cables". An eco-cable is an environmentally benign electric wire/cable that uses

61   environmentally friendly materials and materials with a reduced environmental footprint

62   like polyethylene (PE) and cross-linked polyethylene (XLPE) (Mo et al., 2013).

63   Among the many types of plastics, polyolefin—plastics that have a specific

64   gravity (SG) lower than water (< 1)—are the most popular class of plastics (Plastic waste

65   partnership working group, 2020). These include polypropylene (PP) and polyethylene

66   (including low-density polyethylene (LDPE), linear low-density polyethylene (LLDPE),

67   Medium-density polyethylene (MDPE), and high-density polyethylene (HDPE)), and in

68   2018, PlasticsEurope (2019) reported that PP, LDPE/LLDPE, and HDPE/MDPE

69   constituted 19.3%, 17.5%, and 12.2%, respectively of overall global plastic consumption.

70   XLPE is a variant of PE (usually HDPE) that was modified by using organic

71   peroxides, ionizing radiation or silane compounds to create cross-linking polymeric

72   chains and change PE from being thermoplastic to become thermoset (Bang et al., 2004).

73   XLPE does not melt at 120°C and has better thermal stability, mechanical strength and

74   corrosion resistance compared with PE. Because of its thermosetting property, XLPE

75   cannot be recycled together with PE via conventional melting. XLPE can, however, be

76   recycled via other methods like powdered filler recycling—an approach using

77   pulverization and co-melting with other thermoplastics—to produce filler materials

78   (Bang et al., 2004), supercritical fluid processing method using thermo-plasticizing

79   technology for de-crosslinking (Goto et al., 2011; Tokuda et al., 2003) and pyrolysis (Mo

80   et al., 2013).

81   Because XLPE cannot be recycled together with PE, the separation of these

82   plastics for more efficient recycling is required. Majority of plastic-plastic separation

83   methods used today are based on separation techniques developed in mineral processing

84   (Hori et al., 2009a, 2009b; Ito et al., 2010, 2019a, 2019b, 2020; Phengsaart et al., 2020;

85   Tsunekawa et al., 2005, 2012) while metal separation-extraction strategies for mixed

86   metal-plastic wastes like E-wastes are modified from hydrometallurgy (Calderon et al.,

87   2020; Choi et al., 2020, 2021; Jeon et al., 2018b, 2020a, 2020b) or pyrometallurgy (Inano

88   et al., 2019). Among all separation methods, jig separation—a type of gravity separation

89   that separate the particles based on their motion in fluid—is one of the oldest and most

90   widely used in mineral processing especially for coal cleaning because of its simple

91   operation, low cost and high efficiency (Tsunekawa et al., 2005).

92   The authors have developed the RETAC jig (Fig. 1 (a)) for plastic recycling and

93   modified the RETAC jig into a reverse jig (Fig. 1 (b)) and a hybrid jig (Fig. 1 (c)) (Hori

94   et al., 2009a, 2009b; Ito et al., 2010, 2019a, 2019b, 2020; Tsunekawa et al., 2005, 2012).

95   The reverse jig can separate plastics floating on the water surface (Ito et al., 2010) while

96   the hybrid jig can separate plastics having similar specific gravities by attaching air

97   bubbles selectively onto hydrophobic plastics (Hori et al., 2009b). Using this jig

98   technologies, it is possible to separate several kinds of plastics by incorporating them in

99   various recycling unit operations (Fig. 2). For example, plastics can be separated by sink-

100   float separation using water to recover float and sink products. The sink product (SG >

101   1.0) then goes to either a RETAC jig or a hybrid jig depending on the SGs of plastics

102   (Jeon et al., 2019; Phengsaart et al., 2018, 2020; Tsunekawa et al., 2005). Meanwhile, the

103   floating products (SG < 1.0) can be treated by the reverse jig if the difference in SGs

104   between plastics is substantial. Unfortunately, these previous variants of the RETAC jig

105   cannot treat floating plastics having similar SGs like PE and XLPE.

106   To address this issue, we developed the reverse hybrid jig (Fig. 1 (d)) to separate

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107   floating plastics having similar SGs. In addition, the effects of particle size on separation

108   efficiency and how wetting agents and water pulsation affect bubble attachment during

109   reverse hybrid jig separation were elucidated in this study.
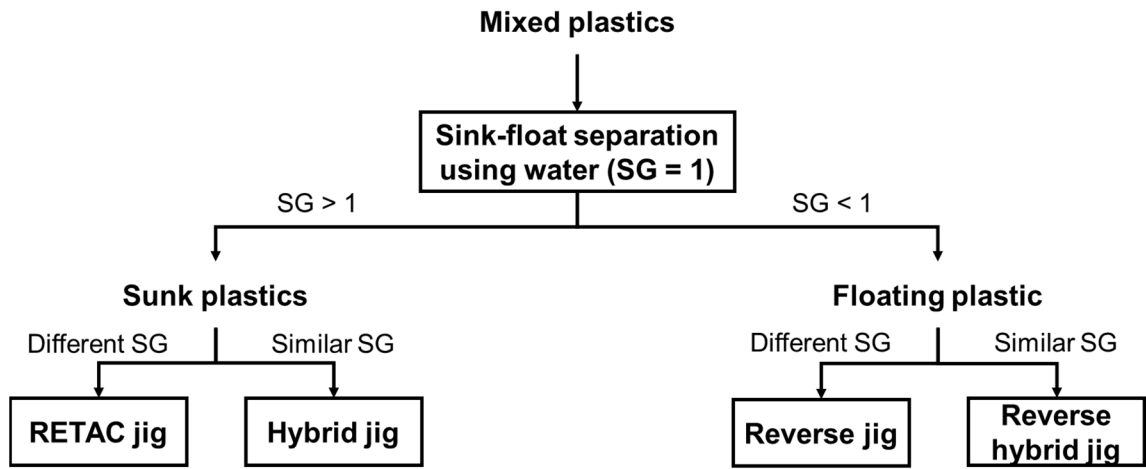
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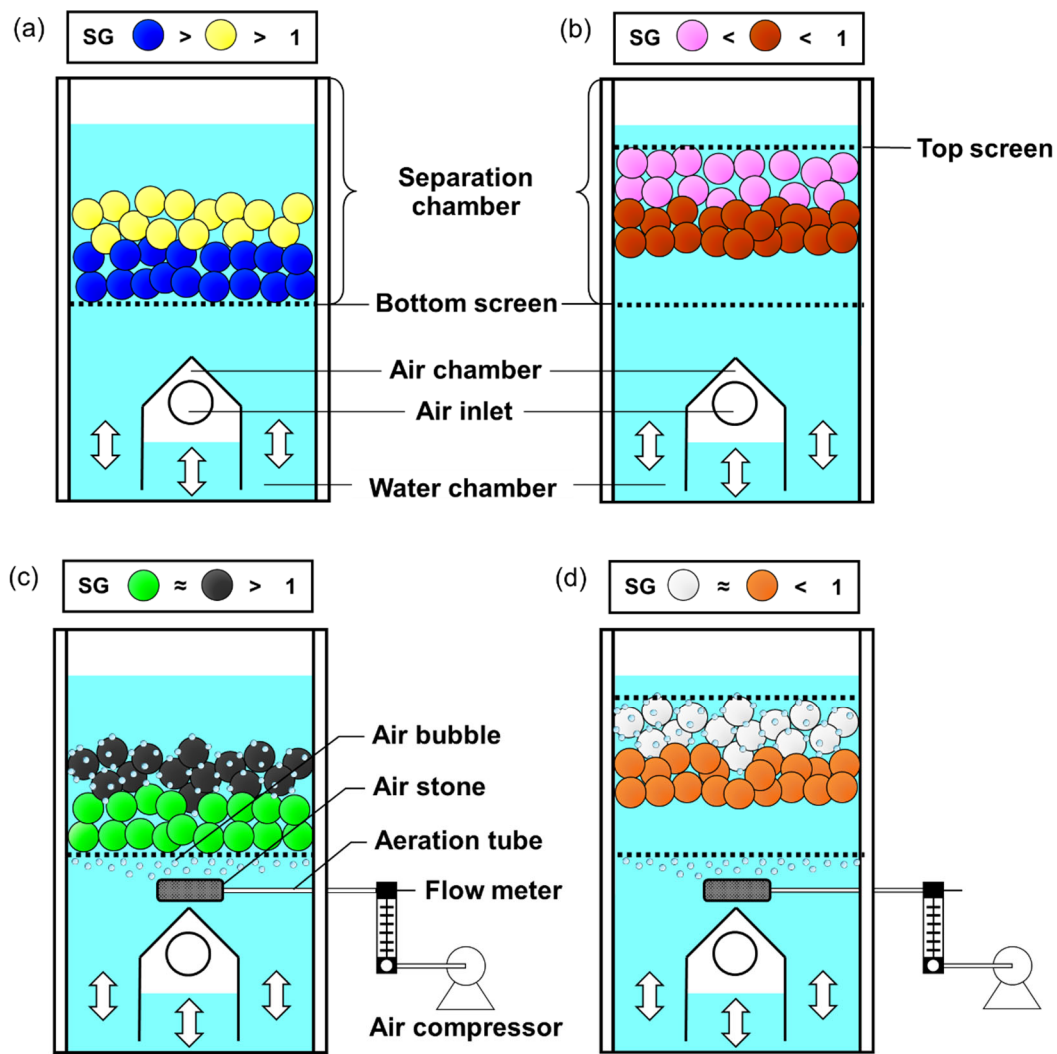
112   Fig. 1. A schematic illustration of (a) RETAC jig, (b) reverse jig, (c) hybrid jig, and (d)

113   reverse hybrid jig.

114

Mixed plastics

Sink-float separation using water (SG = 1)

SG > 1

SG < 1

Sunk plastics

Different SG | Similar SG

RETAC jig | **Hybrid jig**

Floating plastic

Different SG | Similar SG

Reverse jig | **Reverse hybrid jig**

115

116   Fig. 2. Flowchart of plastic separation using advanced jig technology.

117

2.   Outline of the reverse hybrid jig

The TACUB (BATAC) jig is widely used in coal cleaning and the RETAC jig (Fig. 1(a)) is a modified TACUB (BATAC) jig for plastics sinking in water (Tsunekawa et al., 2005). A reverse jig (Fig. 1 (b)) was developed to separate plastics floating on water (Ito et al., 2010). The hybrid jig (Fig. 1 (c))—a combination of jig and flotation technologies—is a modified RETAC jig for separation of hydrophobic and hydrophilic plastics having similar specific gravities by attaching air bubbles selectively onto the hydrophobic plastics (Hori et al., 2009b). In this study, a reverse hybrid jig (Fig. 1 (d)) was developed to separate floating plastics having similar SGs but different wettability.

Fig. 1(d) shows a schematic diagram of the reverse hybrid jig where particles move up and down under the top screen, and the particles are separated based on differences in levitation velocity (Ito et al., 2010). The top screen is made up of a metal mesh (1×1 mm openings). An aeration tube fitted with air stones is installed under the separation chamber and an air pump is connected to the tube. Ragging materials—6 mm diameter alumina balls—were placed on the bottom screen to create uniform water flow and air bubble distribution within the separation chamber. When bubbles attach selectively onto hydrophobic particles, the apparent specific gravity ($SG_{apparent}$) becomes lower and the reverse hybrid jig can potentially separate particles based on the differences

136   in their $SG_{apparent}$ similar to those reported by Ito et al. (2020) for the hybrid jig.

137

3.   Materials and methods

3.1   Samples

Polyethylene (PE, SG = 0.92) and cross-linked polyethylene (XLPE, SG = 0.93) were used in this study and the samples were obtained from an electric wire recycling plant (Kuniosa Metal Co. Ltd., Japan). The samples were crushed by an orient mill (VH16, Seishin Enterprise Co. Ltd., Japan) and sieved to obtain 4 size fractions (+2.8–4.0 mm, +4.0–5.6 mm, +5.6–6.7 mm and +6.7–8.0 mm).

3.2   Reagents

Methyl isobutyl carbinol (MIBC, Wako Pure Chemical Industries Ltd., Japan)— a reagent widely utilized in flotation as a frother to stabilize bubbles in solution—and tannic acid (TA, Wako Pure Chemical Industries, Ltd., Japan)—a wetting agent (sometimes called collector or depressant, depending on its role)—were used in the reverse hybrid jig separation experiments.

3.3   Reverse hybrid jig separation experiments

Reverse hybrid jig separation experiments were carried out under the following conditions: displacement of 20 mm, frequency of water pulsation equal to 30 cycles/min,

156 conditioning time of 5 min (air generation without water pulsation), and separation time

157 of 3 min (air generation with water pulsation). The amounts of samples, water, air flow

158 rate, MIBC dosage and TA dosage are shown in Table 1. After the reverse hybrid jig

159 separation, products were divided into six layers from the top and collected using a

160 vacuum sampling system. Materials in the layers were separated by hand to determine the

161 purity of each layer.

162

163 Table 1. Experimental conditions of the reverse hybrid jig separation.

| Variables	Conditions	164
PE	150 g	
XLPE	150 g	
Water	18 L	
MIBC	20 ppm	
TA	0–500 ppm	
Air flow rate	500 mL/min	
Conditioning time	5 min	
Separation time	3 min	

165

166   **3.4   Measurements of attached-bubble volume**

167   In our previous study, we developed a special laser-assisted measurement setup

168   for the determination of attached-bubble volume on plastic particles during water

169   pulsation to estimate suitable conditions for surface modification (Ito et al., 2020; Fig. 3).

170   In this setup, air bubbles are introduced by a pump under the particle bed, and

171   when bubbles attached to particles, an equivalent water level rise is recorded. This water

172   level rise is accurately measured and recorded by the laser-based level sensor system (IL-

173   S100, Keyence Corporation, Japan), and the attached-bubble volume can then be

174   calculated from changes in water level inside the separation chamber before and after

175   bubble introduction. Measurements of attached-bubble volume were carried out under

176   static and pulsed water conditions (Ito et al., 2020).

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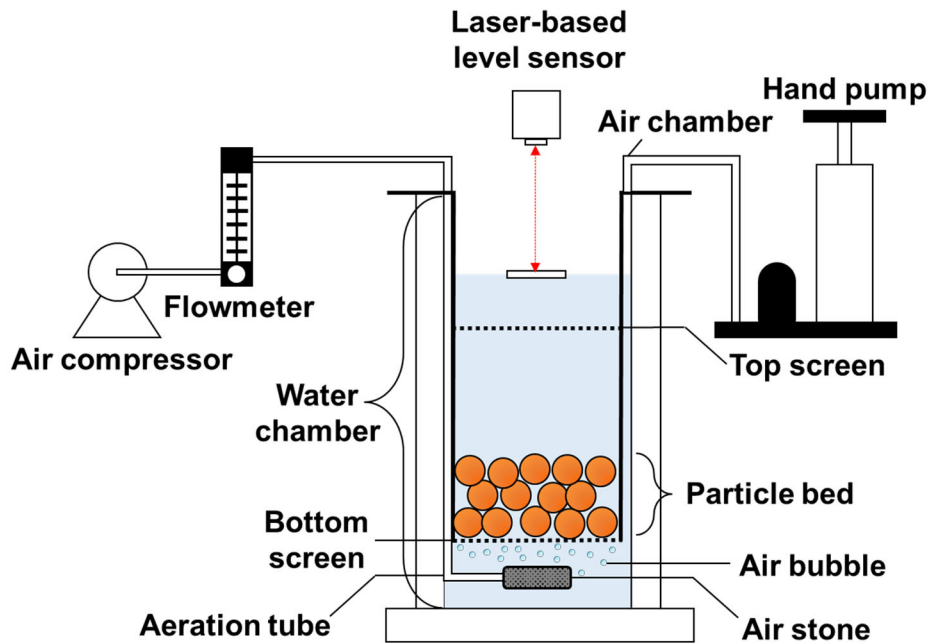
Fig. 3 A schematic diagram of the laser-assisted measurement setup for the determination of attached-bubble volume.

3.4 Surface tension and bubble size measurement

Surface tension of water with different TA dosages was measured using a temperature-controlled reaction vessel connected to a tensiometer (Krüss K100, Krüss GmbH, Germany). Bubble size measurement in reverse hybrid jig separation chamber were carried out at different concentrations of TA and captured using a high-speed digital camera. The captured images were analyzed using an image analysis software (WinRoof v.5, MITANI Corporation, Japan) for 100 bubbles (10 random bubbles from 10 images).

4. Results and discussion

4.1 Effects of bubble attachment on reverse hybrid jig separation

4.1.1 Effects of tannic acid addition

Fig. 4a shows the volume of bubbles attached to the +5.6−8.0 mm PE and XLPE with and without TA. The attached bubble volume measured for XLPE was higher than that of PE regardless of TA concentration and the biggest difference was observed at 200 ppm. The attached bubble volumes to PE and XLPE were also higher at low TA concentrations (50–100 ppm) and decreased with increasing TA concentrations. This suggests that plastics become hydrophilic because of the addition of TA but TA addition caused the sizes of bubbles to increase (Fig. 4b). The bigger bubble size may explain the higher attached bubble volume on the plastic surface (Fig. 4(a)).

Separation tests with a reverse hybrid jig were carried out for mixtures of PE and XLPE (150 g each) under various TA concentrations but the purity of the recovered products was low (XLPE: 54 % as top (1st) layer, PE: 43 % as bottom (6th) layer, TA 250 ppm) (Fig. 5(a)), which could be attributed to excess attached bubble volume on particles. By visual observation during the reverse hybrid jig separation, some of the attached-bubbles detached from particles when water pulsation is applied. To further understand this phenomenon, the effects of water pulsation on bubble detachment behavior from the

15

208   plastic surface was investigated in the next section.
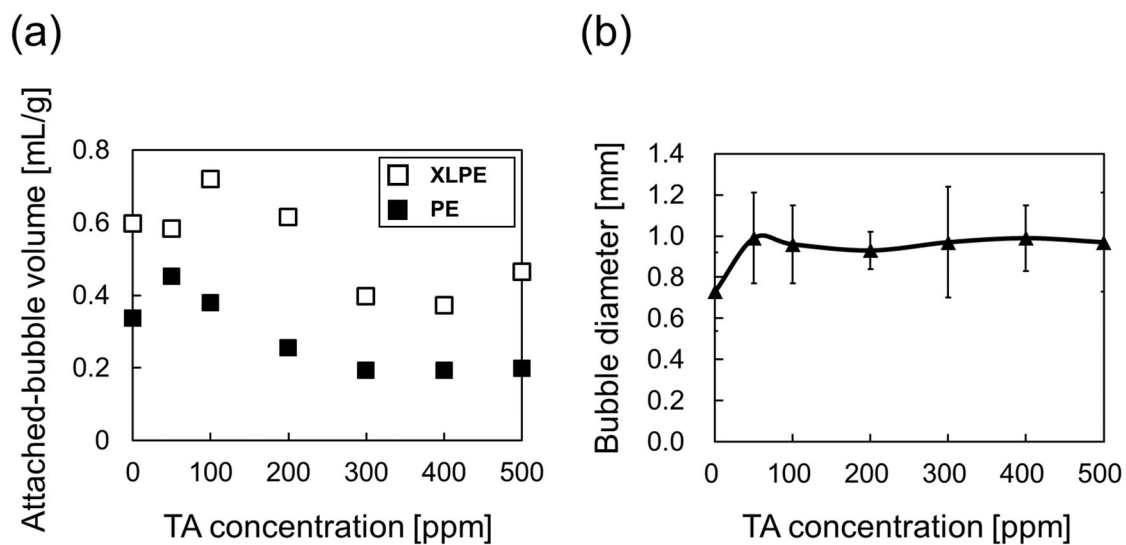
209

210   Fig. 4. (a) attached-bubble volume as a function of TA concentration and (b) bubble

211   diameter as a function of TA concentration.

212

4.1.2   Detachment behavior of attached bubbles and reverse hybrid jig separation

using a two-step approach

The effects of water pulsation on the detachment behavior of bubbles attached to plastic surfaces were investigated using a special laser-assisted measurement setup (Fig. 3). Air bubbles were introduced for 3 minutes to facilitate attachment to plastic particles (Ito et al. 2020). After which, the air pump was turned off and water pulsation was applied using a hand pump and changes in the attached-bubble volume were measured at 250 ppm of TA. The results showed that attached-bubble volume decreased with the number of water pulsation and became constant after 4 pulsations (Fig. 6) and based on these results the $SG_{apparent}$ of XLPE and PE are 0.86 and 0.92, respectively (Ito et al., 2020).

Therefore, reverse hybrid jig separation was carried out using a two-step approach: (i) jigging with air introduction for 5 minutes, and (ii) jigging for 3 minutes without air introduction. Fig. 5 shows the improvement of purity of products using the two-step method (Fig. 5 (b)); that is, 88% XLPE was recovered as top products (1st layer), a 34% improvement from 54% obtained using the one-step approach (Fig. 5 (a)). Similarly, PE purity in bottom products (6th layer) increased from 43% to 74% using the two-step approach. These results suggest that the plastics were separated by differences in bubble detachment behavior and by controlling bubble introduction into the separation

17

chamber, effective separation could be achieved. For the reverse hybrid jig, both

attachment and detachment of bubbles are important parameters. However, the number

of attached bubbles on particles is a function of particle size, so to further understand the

bubble attachment-detachment process, the effects of particle size on reverse hybrid jig

separation was investigated in the next section.
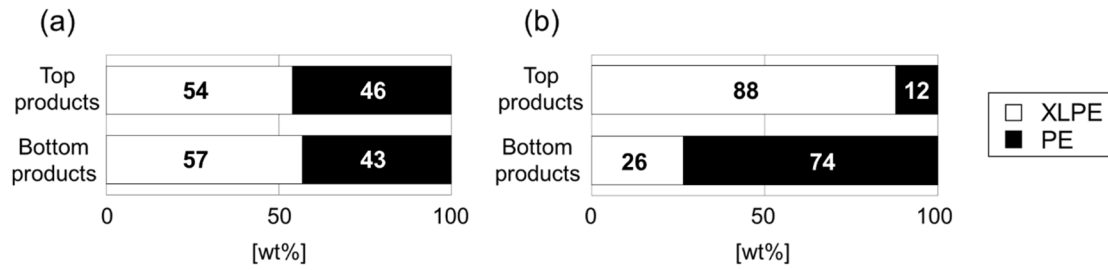
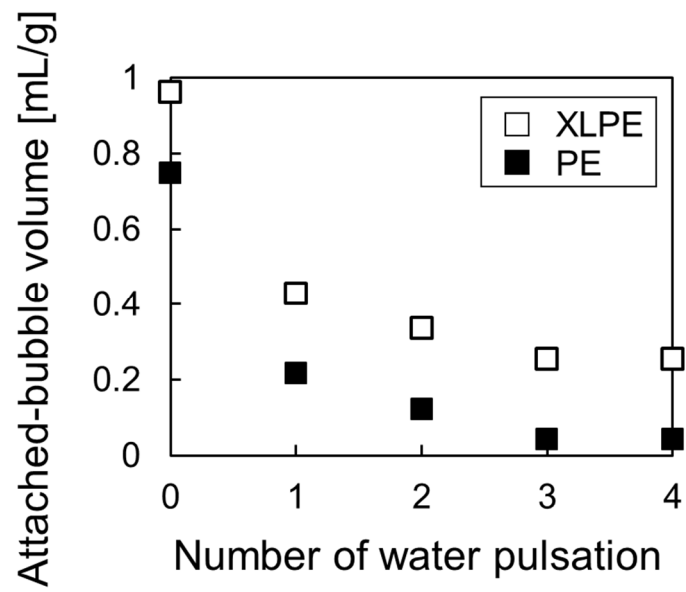Fig. 5 Distribution of XLPE and PE in top (1st layer) and bottom (6th layer) products after

the reverse hybrid jig separation (a) one-step approach (air introduction without jigging

for 5 min and jigging with air introduction for 3 min), and (b) two-step approach (jigging

with air introduction for 5 min and jigging without air introduction for 3 min.).

243

244   Fig. 6 Attached-bubble volume on PE and XLPE as a function of water pulsation without

245   air introduction

246

4.2   Effects of particle size on reverse hybrid jig separation

247

248   Crushed plastic samples were divided into 4 size fractions (+2.8−4.0 mm,

249   +4.0−5.6 mm, +5.6−6.7 mm, and +6.7−8.0 mm) and reverse hybrid jig tests using the

250   one-step approach were carried out for mixtures of PE and XLPE (150 g each) of each

251   size fraction (Fig. 7). The results showed that the purity of XLPE in the top products (1st

252   layer) and the purity of PE in the bottom products (6th layer) increased with increasing

253   size and higher purity products were obtained in the coarser size fraction at a TA

254   concentration of 250 ppm. These results could be explained by the terminal velocity of a

255   particle with attached bubbles. In conventional jig separation, separation occurs because

256   of the difference of particle settling velocities while in the reverse jig (also reverse hybrid

257   jig), separation of particles is facilitated by the difference of their levitation velocities (Ito

258   et al., 2010; Phengsaart et al., 2020). Jig separation (or reverse jig) could achieve better

259   separation efficiency when the difference of terminal settling velocity (or terminal

260   levitation velocity) is larger. During particle settling (or levitation), there is initially no

261   size effect because of the similar initial velocities of particles. With time, however, the

262   particle's acceleration becomes zero and particles reach their terminal velocities, a

263   parameter that is influenced by particle size as shown in Eq. 1 (in laminar flow).

264
$$v_{\infty H}^* - v_{\infty L}^* = (\rho_H - \rho_L)\frac{gD^{*2}}{18\mu}$$
Eq. 1

265   where $v_{\infty H}^{*}$ is the terminal velocity of a heavy particle with spherical shape [m/s]

266           $v_{\infty L}^{*}$ is the terminal velocity of a light particle with spherical shape [m/s]

267   ρ_H is the density of heavy particle [kg/m^3]

268   ρ_L is the density of light particle [kg/m^3]

269   g is the gravitational acceleration [m/s^2]

270   μ is the viscosity of fluid [Pa·s]

271   D^{*} is the equivalent volume diameter [m]

272   Note: the terminal velocity difference ($v_{\infty H}^{*} - v_{\infty L}^{*}$) is larger when the plastic size (D) is

273   coarser.

274   For jig separation with the presence of bubbles (hybrid and reverse hybrid jig),

275   the terminal velocity difference is also influenced by the number of attached bubbles on

276   the particle surface that changes their apparent SGs (Hori et al., 2009b; Ito et al., 2020).

277   Because the average bubble size is around 1 mm (Fig. 4(b)), fewer bubbles could attach

278   to fine particles, so the difference of apparent density becomes small in the fine fraction

279   causing little terminal velocity difference ($v_{\infty H}^{*} - v_{\infty L}^{*}$). This indicates that the terminal

280   velocity difference ($v_{\infty H}^{*} - v_{\infty L}^{*}$) is larger when the plastic size (D) is coarser, which is

281   in line with the results of reverse hybrid jig separation shown in Fig. 7. These results also

282   suggest that the terminal velocity calculated from apparent density is an important

283 parameter for reverse hybrid jig separation.
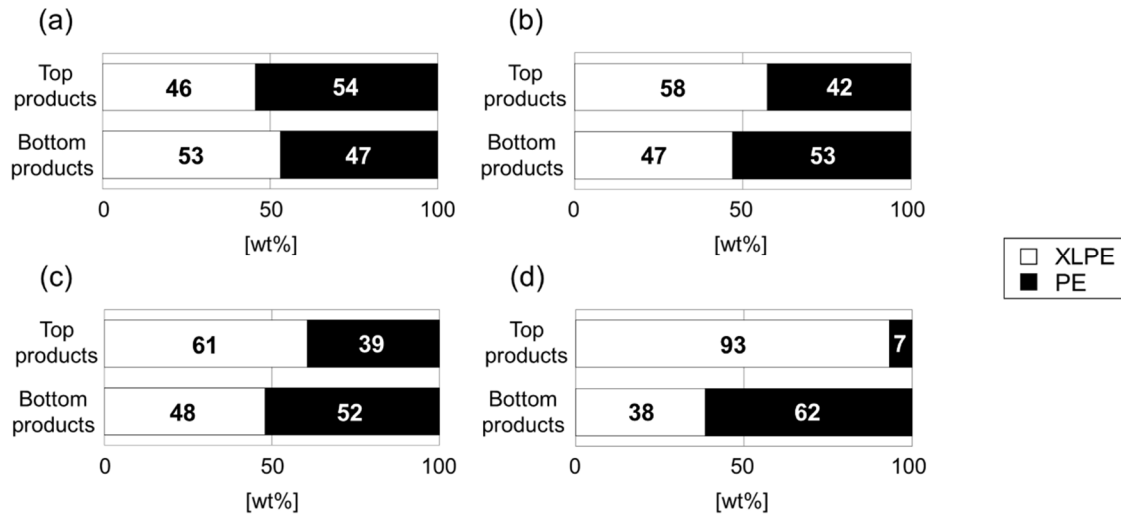
Fig. 7. The distribution of XLPE and PE in top products (1st layer) and bottom products (6th layer) after the reverse hybrid jig separation in 250 ppm TA solution (one-step approach) of PP and XLPE with (a) +2.8−4.0, (b) +4.0−5.6, (c) +5.6−6.7 mm, and (d) +6.7−8.0 mm size fraction.

5. Conclusions

In this study, we developed a novel method to separate floating plastics having similar SGs called the "reverse hybrid jig". The separation of PE and XLPE from waste eco-cable was successfully carried out using this advanced jig separation technique. Moreover, product purity was improved by employing a two-step approach: (1) jigging with air introduction, and (2) jigging without air introduction. The reverse hybrid jig separation efficiency was strongly influenced by both bubble attachment to and detachment from plastic particles. In addition, the effects of particle size and apparent SGs on the levitation velocities and separation efficiency were discussed in line with the results of the reverse hybrid jig separation with different size fractions.

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