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1	Development of the reverse hybrid jig: Separation of polyethylene and cross-linked
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16

## 17 Abstract

18	We have developed various types of jigs for resources recycling focusing
19	primarily on plastic-plastic separation. The RETAC jig could be used to separate plastics
20	heavier than water (specific gravity (SG) > 1.0) with different SGs while the hybrid jig—
21	a process combining jig and flotation-was developed to separate plastics with similar
22	SGs but different surface wettabilities. Meanwhile the reverse jig-a RETAC jig with a
23	top screen—is used to separate plastics lighter than water with different SGs.
24	In this study, a novel method that combines the principle of reverse and hybrid
25	jig, called "reverse hybrid jig separation" was developed to separate plastics lighter than
26	water having similar SGs but different surface wettabilities. The tests were carried out
27	with wastes from an eco-cable wire recycling facility in Japan, which is composed of
28	polyethylene (PE) and cross-linked polyethylene (XLPE). The reverse hybrid jig
29	separation results showed that bubble attachment to and detachment from plastic particles
30	could affect separation efficiency. In addition, the effects of particle size on separation
31	efficiency were also evaluated.
32	

33 **Keywords**: Recycling, jig, reverse hybrid jig, plastic separation, polyolefin

# 35 1. Introduction

36	Plastics have become ubiquitous in our daily life with applications ranging from
37	simple packaging materials to more complicated applications in electronic devices and
38	automobiles (Jeon et al., 2018a; Tabelin et al., 2021a; Thiounn and Smith, 2020). The
39	total production and consumption of plastics, including waste generation, have increased
40	over the last couple of decades (Plastic waste partnership working group, 2020).
41	Fossil fuel-based plastics are further classified into either thermoplastic or
42	thermosets. Thermoplastics are polymers that soften when heated and solidify upon
43	cooling, a property that allows them to be remolded and recycled (i.e., material recycling).
44	In contrast, thermosets are polymers that set into a mold once and cannot be re-softened
45	or remolded again (Plastic waste partnership working group, 2020).
46	There are two common types of plastic recycling: (1) material recycling wherein
47	plastics are recovered and reused, and (2) thermal recycling whereby plastics are used as
48	fuel for power generation. Thermal recycling is more widely used to manage plastic
49	wastes than material recycling. However, some plastics like polyvinyl chloride (PVC),
50	are unsuitable for thermal treatment because they could generate harmful compounds like
51	chlorine gas (Cl2) and dioxins that have adverse effects to industrial processes, the
52	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

peroxides, ionizing radiation or silane compounds to create cross-linking polymeric 71chains and change PE from being thermoplastic to become thermoset (Bang et al., 2004). 72XLPE does not melt at 120°C and has better thermal stability, mechanical strength and 7374corrosion resistance compared with PE. Because of its thermosetting property, XLPE cannot be recycled together with PE via conventional melting. XLPE can, however, be 75recycled via other methods like powdered filler recycling-an approach using 76 pulverization and co-melting with other thermoplastics-to produce filler materials 77 (Bang et al., 2004), supercritical fluid processing method using thermo-plasticizing 78 79 technology for de-crosslinking (Goto et al., 2011; Tokuda et al., 2003) and pyrolysis (Mo et al., 2013). 80

Because XLPE cannot be recycled together with PE, the separation of these 81 82 plastics for more efficient recycling is required. Majority of plastic-plastic separation methods used today are based on separation techniques developed in mineral processing 83 (Hori et al., 2009a, 2009b; Ito et al., 2010, 2019a, 2019b, 2020; Phengsaart et al., 2020; 84 Tsunekawa et al., 2005, 2012) while metal separation-extraction strategies for mixed 85 metal-plastic wastes like E-wastes are modified from hydrometallurgy (Calderon et al., 86 87 2020; Choi et al., 2020, 2021; Jeon et al., 2018b, 2020a, 2020b) or pyrometallurgy (Inano et al., 2019). Among all separation methods, jig separation—a type of gravity separation 88

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

floating plastics having similar SGs. In addition, the effects of particle size on separation
efficiency and how wetting agents and water pulsation affect bubble attachment during
reverse hybrid jig separation were elucidated in this study.

110





112 Fig. 1. A schematic illustration of (a) RETAC jig, (b) reverse jig, (c) hybrid jig, and (d)

113 reverse hybrid jig.





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

# 118 2. Outline of the reverse hybrid jig

119	The TACUB (BATAC) jig is widely used in coal cleaning and the RETAC jig
120	(Fig. 1(a)) is a modified TACUB (BATAC) jig for plastics sinking in water (Tsunekawa
121	et al., 2005). A reverse jig (Fig. 1 (b)) was developed to separate plastics floating on water
122	(Ito et al., 2010). The hybrid jig (Fig. 1 (c))-a combination of jig and flotation
123	technologies—is a modified RETAC jig for separation of hydrophobic and hydrophilic
124	plastics having similar specific gravities by attaching air bubbles selectively onto the
125	hydrophobic plastics (Hori et al., 2009b). In this study, a reverse hybrid jig (Fig. 1 (d))
126	was developed to separate floating plastics having similar SGs but different wettability.
127	Fig. 1(d) shows a schematic diagram of the reverse hybrid jig where particles
128	move up and down under the top screen, and the particles are separated based on
129	differences in levitation velocity (Ito et al., 2010). The top screen is made up of a metal
130	mesh (1×1 mm openings). An aeration tube fitted with air stones is installed under the
131	separation chamber and an air pump is connected to the tube. Ragging materials—6 mm
132	diameter alumina balls-were placed on the bottom screen to create uniform water flow
133	and air bubble distribution within the separation chamber. When bubbles attach
134	selectively onto hydrophobic particles, the apparent specific gravity (SG <sub>apparent</sub> ) becomes

135 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.

## 138 **3.** Materials and methods

# 139 **3.1 Samples**

140	Polyethylene (PE, SG = $0.92$ ) and cross-linked polyethylene (XLPE, SG = $0.93$ )
141	were used in this study and the samples were obtained from an electric wire recycling
142	plant (Kuniosa Metal Co. Ltd., Japan). The samples were crushed by an orient mill
143	(VH16, Seishin Enterprise Co. Ltd., Japan) and sieved to obtain 4 size fractions (+2.8-4.0
144	mm, +4.0–5.6 mm, +5.6–6.7 mm and +6.7–8.0 mm).
145	
146	3.2 Reagents
147	Methyl isobutyl carbinol (MIBC, Wako Pure Chemical Industries Ltd., Japan)-
148	a reagent widely utilized in flotation as a frother to stabilize bubbles in solution-and
149	tannic acid (TA, Wako Pure Chemical Industries, Ltd., Japan)-a wetting agent
150	(sometimes called collector or depressant, depending on its role)-were used in the
151	reverse hybrid jig separation experiments.
152	
153	<b>3.3</b> 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.

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	
ТА	0–500 ppm	
Air flow rate	500 mL/min	
Conditioning time	5 min	
Separation time	3 min	

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

In our previous study, we developed a special laser-assisted measurement setup 167 for the determination of attached-bubble volume on plastic particles during water 168pulsation to estimate suitable conditions for surface modification (Ito et al., 2020; Fig. 3). 169 In this setup, air bubbles are introduced by a pump under the particle bed, and 170 when bubbles attached to particles, an equivalent water level rise is recorded. This water 171172level rise is accurately measured and recorded by the laser-based level sensor system (IL-S100, Keyence Corporation, Japan), and the attached-bubble volume can then be 173174calculated from changes in water level inside the separation chamber before and after bubble introduction. Measurements of attached-bubble volume were carried out under 175static and pulsed water conditions (Ito et al., 2020). 176





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

### **3.4** Surface tension and bubble size measurement

183	Surface tension of water with different TA dosages was measured using a
184	temperature-controlled reaction vessel connected to a tensiometer (Krüss K100, Krüss
185	GmbH, Germany). Bubble size measurement in reverse hybrid jig separation chamber
186	were carried out at different concentrations of TA and captured using a high-speed digital
187	camera. The captured images were analyzed using an image analysis software (WinRoof
188	v.5, MITANI Corporation, Japan) for 100 bubbles (10 random bubbles from 10 images).
189	

#### 190 4. **Results and discussion**

191 4.1 Effects of bubble attachment on reverse hybrid jig separation

192

#### 4.1.1 Effects of tannic acid addition

193 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 194that of PE regardless of TA concentration and the biggest difference was observed at 200 195ppm. The attached bubble volumes to PE and XLPE were also higher at low TA 196 concentrations (50-100 ppm) and decreased with increasing TA concentrations. This 197 198suggests 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 199higher attached bubble volume on the plastic surface (Fig. 4(a)). 200

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 (1<sup>st</sup>) layer, PE: 43 % as bottom (6<sup>th</sup>) 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 attachedbubbles detached from particles when water pulsation is applied. To further understand this phenomenon, the effects of water pulsation on bubble detachment behavior from the 208 plastic surface was investigated in the next section.



209

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

211 diameter as a function of TA concentration.

# **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 215216to 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 217218(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 219220 ppm of TA. The results showed that attached-bubble volume decreased with the number 221of water pulsation and became constant after 4 pulsations (Fig. 6) and based on these results the SG<sub>apparent</sub> of XLPE and PE are 0.86 and 0.92, respectively (Ito et al., 2020). 222

Therefore, reverse hybrid jig separation was carried out using a two-step 223approach: (i) jigging with air introduction for 5 minutes, and (ii) jigging for 3 minutes 224without air introduction. Fig. 5 shows the improvement of purity of products using the 225two-step method (Fig. 5 (b)); that is, 88% XLPE was recovered as top products (1<sup>st</sup> layer), 226a 34% improvement from 54% obtained using the one-step approach (Fig. 5 (a)). 227Similarly, PE purity in bottom products (6<sup>th</sup> layer) increased from 43% to 74% using the 228229two-step approach. These results suggest that the plastics were separated by differences in bubble detachment behavior and by controlling bubble introduction into the separation 230

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 (1<sup>st</sup> layer) and bottom (6<sup>th</sup> 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.).



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

245 air introduction

#### 4.2 Effects of particle size on reverse hybrid jig separation

Crushed plastic samples were divided into 4 size fractions (+2.8-4.0 mm, 248+4.0-5.6 mm, +5.6-6.7 mm, and +6.7-8.0 mm) and reverse hybrid jig tests using the 249250one-step approach were carried out for mixtures of PE and XLPE (150 g each) of each size fraction (Fig. 7). The results showed that the purity of XLPE in the top products (1<sup>st</sup> 251layer) and the purity of PE in the bottom products (6<sup>th</sup> layer) increased with increasing 252253size and higher purity products were obtained in the coarser size fraction at a TA concentration of 250 ppm. These results could be explained by the terminal velocity of a 254particle with attached bubbles. In conventional jig separation, separation occurs because 255of the difference of particle settling velocities while in the reverse jig (also reverse hybrid 256jig), separation of particles is facilitated by the difference of their levitation velocities (Ito 257et al., 2010; Phengsaart et al., 2020). Jig separation (or reverse jig) could achieve better 258separation efficiency when the difference of terminal settling velocity (or terminal 259levitation velocity) is larger. During particle settling (or levitation), there is initially no 260size effect because of the similar initial velocities of particles. With time, however, the 261particle's acceleration becomes zero and particles reach their terminal velocities, a 262263parameter that is influenced by particle size as shown in Eq. 1 (in laminar flow).

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

265	where	$\mathcal{V}_{\infty H}^{*}$	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		ρн	is the density of heavy particle [kg/m <sup>3</sup> ]
268		$ ho_L$	is the density of light particle [kg/m <sup>3</sup> ]
269		g	is the gravitational acceleration [m/s <sup>2</sup> ]
270		μ	is the viscosity of fluid [Pa·s]
271		$D^{*}$	is the equivalent volume diameter [m]
272	Note: tl	he termir	hal velocity difference $(v_{\infty H} * - v_{\infty L} *)$ is larger when the plastic size (D) is
273	coarser		
274		For iig	separation with the presence of bubbles (hybrid and reverse hybrid iig).

jig separation with the presence of bubbles (hybrid and reverse hybrid jig), the terminal velocity difference is also influenced by the number of attached bubbles on 275the particle surface that changes their apparent SGs (Hori et al., 2009b; Ito et al., 2020). 276Because the average bubble size is around 1 mm (Fig. 4(b)), fewer bubbles could attach 277to fine particles, so the difference of apparent density becomes small in the fine fraction 278causing little terminal velocity difference  $(v_{\infty H}^* - v_{\infty L}^*)$ . This indicates that the terminal 279velocity difference  $(v_{\infty H}^* - v_{\infty L}^*)$  is larger when the plastic size (D) is coarser, which is 280281in line with the results of reverse hybrid jig separation shown in Fig. 7. These results also suggest that the terminal velocity calculated from apparent density is an important 282



283 parameter for reverse hybrid jig separation.

Fig. 7. The distribution of XLPE and PE in top products (1<sup>st</sup> layer) and bottom products

286 (6<sup>th</sup> layer) after the reverse hybrid jig separation in 250 ppm TA solution (one-step

- 287 approach) of PP and XLPE with (a) +2.8-4.0, (b) +4.0-5.6, (c) +5.6-6.7 mm, and (d)
- 288 +6.7-8.0 mm size fraction.

#### 290 **5. Conclusions**

In this study, we developed a novel method to separate floating plastics having 291similar SGs called the "reverse hybrid jig". The separation of PE and XLPE from waste 292eco-cable was successfully carried out using this advanced jig separation technique. 293Moreover, product purity was improved by employing a two-step approach: (1) jigging 294with air introduction, and (2) jigging without air introduction. The reverse hybrid jig 295296separation efficiency was strongly influenced by both bubble attachment to and detachment from plastic particles. In addition, the effects of particle size and apparent 297298SGs on the levitation velocities and separation efficiency were discussed in line with the results of the reverse hybrid jig separation with different size fractions. 299

300

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