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1 **ORIGINALARTICLE**

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3 **Improvement of hybrid jig separation efficiency using wetting agents for the recycling of**
4 **mixed-plastic wastes**

5
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14
15 **Abstract**

16
17 We have developed the hybrid jig which combines the principles of jig separation and flotation. However,
18 the selectivity of bubble attachment in water was poor because most plastic have inherently hydrophobic
19 surfaces, so development of surface modification techniques for plastic particles would expand its
20 application to the material recycling of plastics.

21 In this study, the hybrid jig separation of the polypropylene with glass fiber (PPGF) and high
22 impact polystyrene (HIPS) having similar SG and surface wettability were investigated with three wetting
23 agents (Di-2-ethylhexyl sodium sulfosuccinate (Aerosol OT, AOT), sodium lignin sulfonate (NaLS), and
24 tannic acid (TA)). The results showed that the probability of bubble attachment was influenced by wetting
25 agents because of their strong effects on the surface tension of solution and surface wettability of plastics.
26 These results suggest that wetting agents could be utilized to control the selectivity of bubble attachment
27 and improve the hybrid jig separation efficiency. In addition, since the hybrid jig separation of polyvinyl
28 chloride (PVC) and polyamide, nylon-66 (PA) using AOT was imperfect, a two-step approach, composed
29 of a pre-wetting step (first step) in a solution containing the wetting agent (AOT) and hybrid jig
30 separation in water (second step), was proposed.

31
32 **Keywords:** Recycling, jig, hybrid jig, plastic, wetting agent

34 **Introduction**

35 The total global production of plastics has grown from around 1.5 million tons in 1950 to 348 million
36 tons in 2017 and worldwide consumption has been increasing annually at a rate of 5% with the largest
37 increase reported in Asia [1, 2]. There are two common ways to recycle plastics: (i) material recycling
38 whereby plastic wastes are recovered for the production of new plastics, and (ii) thermal recycling
39 whereby plastic wastes are used as fuel for power generation. Between the two, material recycling is more
40 sustainable and profitable but requires exceptionally efficient separation of different types of plastics from
41 mixed-plastic wastes to obtain products with very high purities (>99.9%). Achieving this very high plastic
42 purity in recycling is very difficult, so mixed-plastic wastes are more commonly used in thermal recycling.
43 In recent years, however, several studies have utilized mineral processing techniques to separate
44 mixed-plastic wastes into their individual components to facilitate material recycling (e.g., gravity
45 separation [3-10], dense medium separation [11-13], electrical separation [14-16], flotation [12, 17-28]).

46 Flotation is one of the common mineral processing techniques for fine fraction (minerals: -75
47 μm ; coal: -150 μm) since normally, fine grinding is required (μm) for ores to achieve sufficient liberation
48 of target minerals [9, 29]. Most of minerals have hydrophilic surface so, a collector (e.g., xanthate,
49 aerofloats [30]) is usually added to selectively change the surface wettability to enhance the separation
50 efficiency. In contrast, plastic flotation is usually carried out with wetting agents (e.g., AOT [22], NaLS
51 [22], CaLS [12, 22-24], TA [22, 25, 26], PVA [22, 26-28]) since most plastic have inherently hydrophobic
52 surfaces. However, for resources recycling, especially plastics, sufficient liberation is already achieved in

53 even at coarser particle sizes (mm to cm) [30-32]. Moreover, additional size reduction (i.e., crushing and
54 grinding) required for flotation that incurs higher costs and energy is needed [30].

55 One potentially effective technique for the separation of coarser size of plastics from
56 mixed-plastic wastes is the use of jigs. Jigs can separate the particles at coarse size fractions (+0.5 mm)
57 and jig is a gravity concentrator that separate the particles based on differences in their densities or
58 specific gravities (SG) [7, 30, 33]. Among the various types of jigs, the TACUB (Takakuwa air chamber
59 under bed) jig, commercially marketed as BATAAC jig, is a popular and commonly used type of jig
60 especially in coal cleaning because of its more uniform pressure distribution inside the separation
61 chamber that provides higher separation efficiency. This uniform pressure distribution is facilitated by a
62 series of pneumatically operated multiple air chambers, usually two cells under the separation chamber,
63 that extend to its full width. Pressurized air is injected into the air chambers, causing water within the
64 separation chamber to pulsate and induce stratification [3, 6, 30].

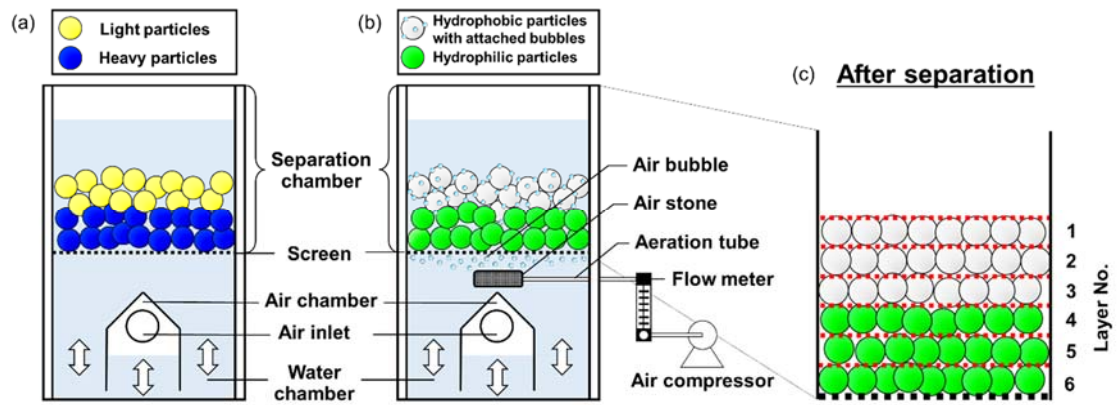
65 The TACUB jig was, however, ineffective for mixed-plastic wastes because SG differences of
66 the various types of plastics in mixed-plastic wastes are very small. To address this problem, Tsunekawa
67 et al. [3] developed the RETAC jig (R&E, Co., Ltd., Japan), an improved version of the TACUB jig, that
68 facilitates plastic-plastic separation via precision control of the wave form during jig separation (Fig. 1(a))
69 [3, 4]. The RETAC jig, for example, successfully separated polystyrene (PS), acrylonitrile butadiene
70 styrene (ABS), and polyethylene terephthalate (PET) from crushed copy machines [3]. Although
71 effective, separation efficiency of the RETAC jig dramatically decreases when the density difference of

72 plastics to be separated becomes small. Unfortunately, many types of plastics developed in recent years
73 for identical applications have very similar SG. For example, two types of plastics used in the
74 manufacture of large home appliances, polypropylene with glass fiber (PPGF) and high impact
75 polystyrene (HIPS), both have SG equal to 1.04. In theory, separation of materials with similar SG is
76 impossible using gravity separation techniques like jigs.

77 A new type of jig called the hybrid jig was developed to address this limitation. The hybrid jig
78 works by combining the principles of gravity separation and flotation and could separate materials with
79 similar SG so long as their surface wettability properties are different (Fig. 1(b)) [5]. Separation occurs
80 due to the attachment of bubbles to more hydrophobic plastic particles that make them lighter than the
81 less hydrophobic plastic particles that causes stratification during water pulsation.

82 In a previous study of the authors, hybrid jig separation of pure plastic mixtures (i.e.,
83 polyethylene (PE), polyethylene terephthalate (PET), polyvinyl chloride (PVC)) in water was carried out
84 and good separation efficiency was reported [5]. However, the selectivity of bubble attachment in water
85 for other plastics was poor because most plastic have inherently hydrophobic surfaces, so development of
86 surface modification techniques for plastic particles would expand its application to the material recycling
87 of plastics. This means that surface modification of plastics is required for the improvement of
88 mixed-plastic waste separation using the hybrid jig, so in this study, the effects of wetting agents on
89 bubble attachment probability and hybrid jig separation efficiency were investigated. In addition, a
90 two-step approach to improve hybrid jig separation efficiency using wetting agents is proposed.

91



92

93 **Fig. 1** Schematic illustrations of (a) RETAC jig, (b) Hybrid jig, and (c) how the products were partitioned

94 into 6 layers after separation.

95

96 **Materials**

97

98 **Samples**

99 Polypropylene with glass fiber (PPGF) and high impact polystyrene (HIPS) obtained from a recycling
100 facility of home appliances in Japan, and virgin plastic boards of polyvinyl chloride (PVC) and polyamide,
101 Nylon-66 (PA) were used in this study and the SG of these samples are listed in Table 1. The virgin
102 plastic boards were crushed by an orient mill (VH16, Seishin Enterprise Co. Ltd, Japan) and screened to
103 obtain suitable size fractions for the hybrid jig separation experiments.

104

105 Table 1. Plastic samples and their specific gravities (SG).

Samples	Abbreviation	Specific gravity
Polypropylene with glass fiber	PPGF	1.04
High impact polystyrene	HIPS	
Polyvinyl chloride	PVC	1.38
Polyamide, Nylon-66	PA	1.37

106

107

108 **Reagents**

109 Methyl isobutyl carbinol (MIBC, Wako Pure Chemical Industries Ltd., Japan), a reagent widely utilized
110 in flotation to stabilize bubble formation in solution, was used in the hybrid jig separation experiments.
111 Three types of wetting agents were evaluated in this study: (1) Di-2-ethylhexyl sodium sulfosuccinate
112 (Aerosol OT, AOT, Wako Pure Chemical Industries Ltd., Japan), (2) sodium lignin sulfonate (NaLS,
113 Tokyo Chemical Industry Co. Ltd., Japan), and (3) tannic acid (TA, Wako Pure Chemical Industries, Ltd.,
114 Japan).

115 **Experimental methods**

116

117 **Surface tension and contact angle measurements**

118 Surface tension of solutions containing each of the wetting agents was measured using a

119 temperature-controlled reaction vessel connected to a tensiometer (Krüss K100, Krüss GmbH, Germany).

120 For the contact angle measurements, an air bubble generated from a syringe needle was introduced

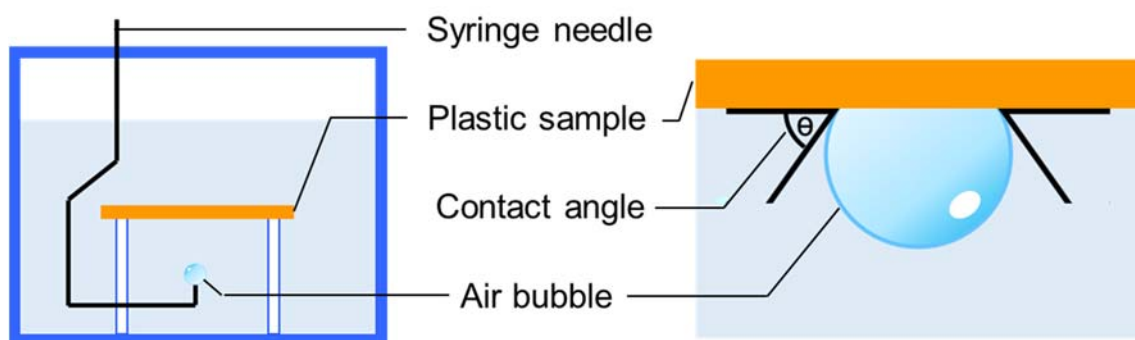
121 underneath the plastic sample submerged in solutions containing each of the wetting agents and the

122 contact angle (angles between particle surface (solid-liquid interface) and bubble surface (gas-liquid

123 interface)) (Fig. 2) was measured using a high-resolution digital microscope with image analysis

124 capability (VHX-1000, Keyence Corporation, Japan).

125



126

127

128 **Fig. 2** Schematic diagrams of the contact angle measurement

129

130 **Adsorption and desorption experiments**

131 To obtain adsorption isotherm of AOT on PA [34], the adsorption experiments of AOT on PA were carried
132 out. The particles of PA (25 g) were stirred (400 rpm) in to AOT solution (30, 50, 80, 100, 150, 180, 200,
133 and 250 ppm) for 10 min then, left for 120 min. The dissolved sulfur concentration of remaining solution
134 was measured by inductively coupled plasma atomic emission spectroscopy (ICP-AES, ICPE-9820,
135 Shimadzu Corporation, Japan) (margin of error = $\pm 2\%$).

136 For desorption experiments, PA particles were put in 10 ppm AOT solution for 3 min and then,
137 washed by water. The particles were then put in another beaker filled with water and the dissolved sulfur
138 concentration of solution was measured by ICP-AES after 30, 60, 90, and 120 min to calculate the
139 desorption amount of AOT from PA.

140

141 **Hybrid jig separation experiments**

142 Fig. 1(b) shows a schematic diagram of the desktop hybrid jig separation apparatus used in this study.

143 This device is a modified desktop RETAC jig that has additional aeration tubes fitted with air stones
144 installed under the separation chamber (145 mm long, 155 mm wide and 320 mm high) to generate air
145 bubbles. The hybrid jig separation experiments were done by putting plastic samples and a solution (20 L)
146 containing water, MIBC, and one of the wetting agents into the separation chamber (Table 2). Air bubbles
147 were introduced underneath the screen simultaneously as water pulsation begun (Fig. 1(b)). Table 2
148 summarizes the conditions of hybrid jig separation experiments conducted in this study. The water

149 pulsation was decided based on the research of Hori et. al., 2009 [4]. After each run, the products were
 150 divided from the top into six layers (Fig. 1(c)) and then collected using a vacuum sampling system.
 151 Different plastics in each layer were separated by hand-picking to measure the purity of products.

152

153 Table 2. Conditions of the hybrid jig separation tests.

	Experiment 1	Experiment 2
	HIPS/PPGF	PVC/PA
Weight of sample [g]	250	
Particle size [mm]	+3.0 - 8.0	
Displacement [mm]	10	
Frequency of water pulsation [cycle/min]	30	
Separation time [min]	3	
Air rates [L/min]	1.5	
Concentration of stabilize bubble formation reagent [ppm or mg/L]	20	
Concentration of wetting agent [ppm or mg/L]	350 (NaLS, TA)	10 (AOT)

154

155

156 **Results and discussion**

157

158 **Separation of PPGF and HIPS using bubble attachment probability**

159 PPGF is typically used for drum-type washing machines while HIPS is a common type of plastic used in
160 the manufacture of many home appliances because of its durability. Although the SG of PP is less than 1.0,
161 addition of glass-fiber to create the stronger PPGF increased its SG to 1.04, which is equal to that of HIPS.
162 Because both of these types of plastics are common components of large home appliances, they are mixed
163 during recycling and should be separated to improve material recycling efficiency.

164 Preliminary experiments to separate PPGF and HIPS using the hybrid jig without any wetting
165 agent showed that separation was impossible because of the indiscriminate attachment of bubbles on both
166 types of plastics. Fig. 3(a) shows the hybrid jig experimental results for the PPGF-HIPS mixture (3.0 to
167 8.0 mm) in solutions containing 350 ppm of AOT, NaLS or TA. Purity in the top (1st) and bottom (6th)
168 layers (Fig. 1(c)) with AOT (Fig. 3(a-1)) was lower than those with NaLS (Fig. 3(a-2)) and TA (Fig.
169 3(a-3)). With NaLS, the PPGF purity in the top layer and the HIPS purity in the bottom layer were 97%
170 and 99%, respectively and with TA, the PPGF purity in the top layer and the HIPS purity in the bottom
171 layer were 99% and 86%, respectively. These results suggest that separation of both plastics is possible,
172 and the type of wetting agent and their concentrations are important parameters to improve the hybrid jig
173 separation efficiency.

174 The effects of a wetting agent on particle-bubble interactions depend on tensile forces acting on

175 the particle that lead to the development of an angle between the particle surface and the bubble surface
176 [30]. At equilibrium

$$177 \quad \gamma_{s/a} = \gamma_{s/w} + \gamma_{w/a} \cos \theta \quad (1)$$

178 where $\gamma_{s/a}$, $\gamma_{s/w}$, and $\gamma_{w/a}$ are the surface energies between solid and air, solid and water, and water and air,
179 respectively, and θ is the contact angle (angles between particle surface (solid-liquid interface) and bubble
180 surface (gas-liquid interface)) (Fig. 1). The attachment of bubbles on the surface of particles is strongly
181 dependent on the work of adhesion ($W_{s/a}$), which is defined as the force required to break the
182 particle-bubble interface and is equal to the work needed to separate the solid-air interface and produce
183 separate solid-air, air-water and solid-water interfaces (Eq. 2) [30].

$$184 \quad W_{s/a} = \gamma_{w/a} (1 - \cos \theta) \quad (2)$$

185 The work of adhesion is further influenced by the surface wettability of the particle, determined
186 by the contact angle (θ), and the surface tension of solution. More hydrophobic (i.e., larger contact angle)
187 particles have larger work of adhesion when in contact with bubbles. Similarly, solutions with higher
188 surface tension ($\gamma_{w/a}$) increase the work of adhesion between particle and bubble. Both the surface tension
189 and surface wettability of particles could be changed by adding surface acting agents called surfactants
190 [30], so measurements of the contact angles of PPGF and HIPS as well as the surface tension of solutions
191 containing each of the wetting agents were carried out.

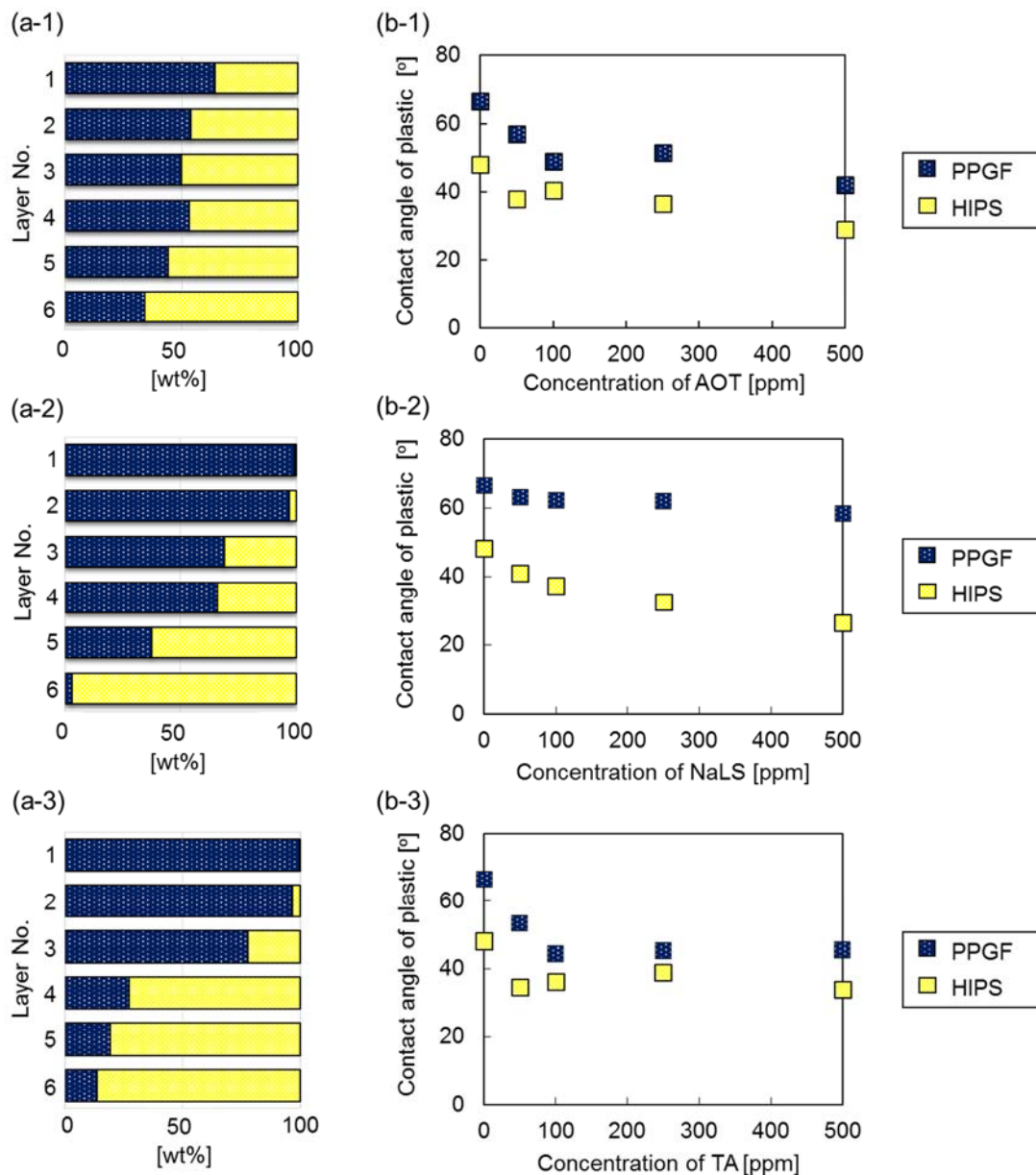
192 Figs. 3(b) and 4 show the contact angles of PPGF and HIPS as well as the surface tension of
193 solutions containing the wetting agents (i.e., AOT, NaLS or TA). The results showed that PPGF is more

194 hydrophobic than HIPS regardless of the type and concentration of wetting agent (Fig. 3(b)).

195 Among the three wetting agents, only AOT lowered the surface tension of solution while the
196 effects of NaLS or TA on it were negligible (Fig. 4). The lower surface tension of solution in the presence
197 of AOT decreased the work of adhesion, resulting in fewer bubble attachments on the particle surface. In
198 comparison, NaLS or TA had negligible effects on the surface tension but decreased the contact angle at
199 higher concentrations (Fig. 4). These results suggest that the work of adhesion and bubble attachment
200 probability decreased in the presence of either NaLS or TA likely because of the adsorption of these two
201 wetting agents onto the surface of plastic particles that made them more hydrophilic. Similarly, the
202 contact angles on both plastics decreased in the presence of AOT (Fig. 3(b-1)) but because this wetting
203 agent also lowered the surface tension of solution (Fig. 4), the net effect was the limited bubble
204 attachment that resulted in low separation efficiency even though both PPGF and HDPS became more
205 hydrophilic (Fig. 3(a-1)). In the case of NaLS, the change in contact angle of HIPS was more dramatic
206 than that of PPGF, which caused selective bubble attachment onto PPGF making it lighter than HIPS that
207 facilitated better stratification during hybrid jig separation (Figs. 3(a-2) and 3(b-2)). In the case of TA, the
208 contact angle of PPGF decreased with increasing TA concentration (Fig. 3(b-3)). However, the contact
209 angle with TA was slightly higher than that with AOT, which likely caused selective bubble attachment on
210 the PPGF surface. These results show that the control of surface characteristics by wetting agents could
211 improve the separation efficiency of the hybrid jig. In the TA solution, similar results were obtained
212 compared with that in NaLS even though the contact angles of HIPS and PPGF were similar. This

213 phenomenon remains unclear because of the difficulty in quantifying the amounts of bubbles attached on
214 plastic particles. Many previous papers reported the interaction between air bubbles and plastic particles
215 during flotation [35, 36] however, the particle and water movement during hybrid jig separation and
216 flotation are different. This means that a reliable measurement technique to quantify bubble attachment
217 volume during hybrid jig separation (with water pulsation) should be developed.

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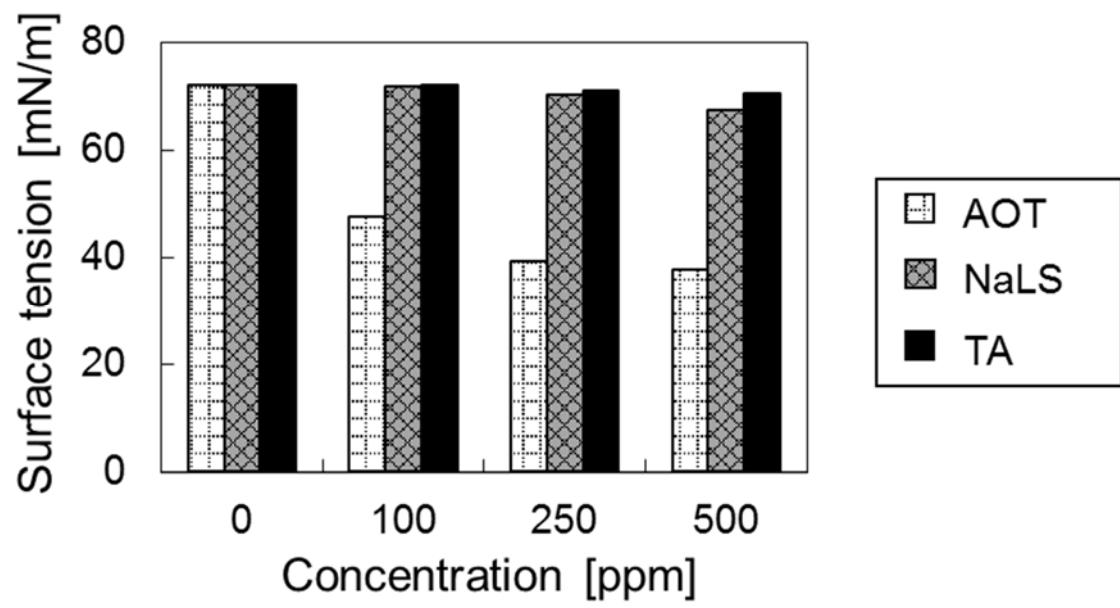
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220 **Fig. 3** (a) The proportion of PPGF and HIPS in each layer after hybrid jig separation tests with (a-1) AOT

221 (350 ppm), (a-2) NaLS (350 ppm), and (a-3) TA (350 ppm) as wetting agents, and (b) contact angles of

222 PPGF and HIPS in (b-1) AOT, (b-2) NaLS, and (b-3) TA solutions

223



224

225 **Fig. 4** Surface tension of solutions containing each of the wetting agents at various concentrations

226

227 **Separation of PVC and PA using a two-step approach**

228 PVC is a common type of plastic used for a variety of applications such as in building and construction,
229 packaging, electrical and electronics, and automobile while PA (or nylon) is an engineering plastic used in
230 clothing, electrical and electronic, and automobile [1]. These types of plastics can be found together in
231 waste electrical and electronic equipment (WEEE) and automotive shredder residue (ASR) from end of
232 life vehicle (ELV) and due to their similar SG (1.38 and 1.37), in this study hybrid jig was applied to
233 separate them into individual components to improve material recycling efficiency.

234 The results from the previous section showed that wettability control by selective bubble
235 attachment with the aid of wetting agents could improve the hybrid jig separation efficiency. In this
236 section, separation of PVC and PA using a two-step approach was investigated. Hybrid jig tests of PVC
237 (SG = 1.38) and PA (SG = 1.37) were carried out in water containing 10 ppm of AOT. The results showed
238 that these two plastics could not be separated without AOT (i.e., separation efficiency was almost 0%)
239 because of the indiscriminate attachment of bubbles to both types of plastics. Fig. 5(a-1) shows the result
240 of hybrid jig separation with 10 ppm of AOT. The PVC purity in the top layer and the PA purity in the
241 bottom layer were 84% and 98%, respectively. Separation of PVC and PA occurred because AOT lowered
242 the contact angle of PA but not that of PVC as shown in Fig. 5(b). However, bubble attachment
243 probability on PVC slightly decreased with AOT addition (Fig. 5(b)) due to decrease of surface tension as
244 discussed earlier (Fig. 4), that likely caused imperfect separation (Fig. 5(a-1)).

245 To improve the separation efficiency, a two-step hybrid jig method (pre-wetting of PA) is

246 proposed. In the first step, plastic mixture of PVC and PA are put in 10 ppm AOT solution for 3 min.

247 Then, solution is discharged, and water is filled instead (second step) and hybrid jig separation in water is

248 carried out. Fig. 5(a-2) shows that higher purities of PVC (94%) in the top layer and PA (99%) in the

249 bottom layers were obtained compared with the single-step approach because higher surface tension of

250 second step method than one-step method caused better bubble attachment on PVC (Fig. 5(a-1)).

251 However, due to the replacement of water in second step may cause some effect thus, effects of time after

252 water replacement was checked. The results showed that hybrid jig separation just after water

253 replacement was effective (Fig. 5(a-2)) however, several tens of minutes after water replacement showed

254 worse hybrid jig separation efficiency because bubble attachment to the PVC became difficult again. This

255 lower bubble attachment probability of PVC may cause by the decrease of surface tension due to the

256 detachment of AOT from PA surface. To understand the attachment and detachment behaviors of AOT on

257 PA, the adsorption and desorption experiments were carried out. The adsorption of AOT on PA was

258 confirmed (Fig. 6(a)) and desorption of AOT from PA with time (Fig. 6(b)) showed that AOT was readily

259 desorbed causing the surface tension of solution to decrease with time (Fig. 6(b)). In other words, the

260 separation results become bad with time because the wettability of PVC was enhanced due to the decrease

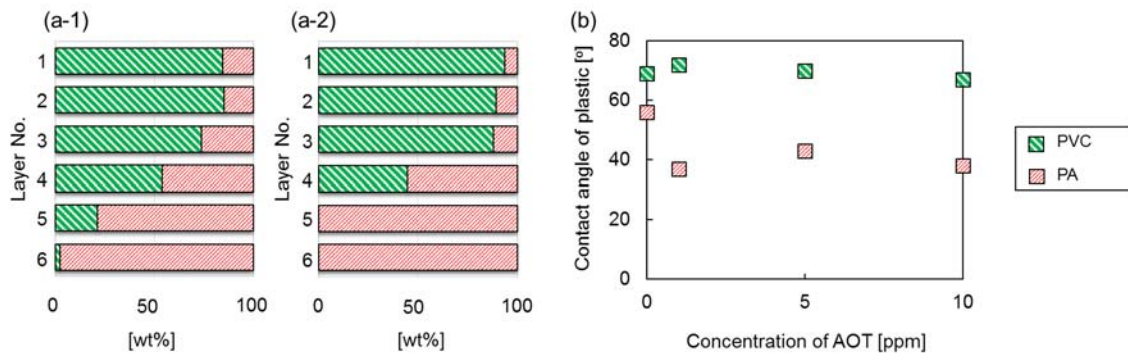
261 in surface tension of solution caused by the desorption AOT adsorbed on PA during the pre-wetting stage.

262 This means that although pre-wetting was effective, the adsorption-desorption behavior of wetting agent

263 could strongly affect the separation efficiency, so precise control of the wettability of samples is required.

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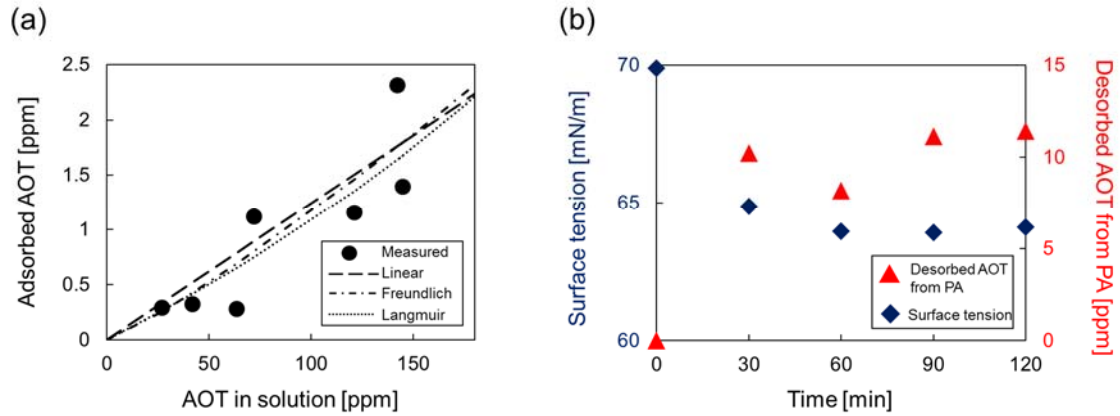
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267 **Fig. 5** (a) The proportion of PVC and PA in each layer after hybrid jig separation tests (a-1) in AOT (10

268 ppm) solution (one-step method) and (a-2) after solution replacement with water (two-step approach), and

269 (b) contact angles of PVC and PA in the AOT solutions

270



271

272

273 **Fig. 6** (a) Adsorption characteristics of AOT onto PA with linear, Freundlich and Langmuir isotherms, and

274 (b) desorbed AOT concentration from PA with time and the corresponding changes in the surface tension

275 of the solution

276

277 **Conclusions**

278

279 This paper evaluated the improvement of hybrid jig separation of mixed-plastic wastes using wetting
280 agents and the findings of this study are summarized as follows:

281 • Wetting agents could change the surface tension of solutions by changing the water-air surface
282 properties and the surface wettability (contact angle) of plastics by surface adsorption, both of which
283 lowered the bubble attachment probability.

284 • For the separation of PPGF and HIPS, surface modification using wetting agents (i.e., AOT, NaLS
285 or TA) could improve the separation efficiency.

286 • In the case of PPGF/HIPS with AOT, the changes in surface tension and the decrease of contact
287 angles of both plastics caused lower bubble attachment probability, so selective attachment of
288 bubbles was limited that caused lower separation efficiency.

289 • In the case of PPGF/HIPS with NaLS, the changes in surface tension were negligible and the contact
290 angle on HIPS decreased while that on PPGF was unchanged, so selective attachment of bubbles on
291 PPGF occurred and higher separation efficiency was obtained.

292 • In the case of PVC/PA with AOT, the surface tension decreased and the contact angle on PA
293 decreased while that of PVC did not change, which caused selective attachment of bubbles on PVC.

294 • A two-step approach could increase the surface tension, increase the bubble attachment probability
295 on PVC, and dramatically improved the hybrid jig separation efficiency.

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300

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