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Industrial Ramie growing on reclaimed ion-adsorption Rare Earth Elements mine tailings in Southern China: Defibrillation and fibers quality

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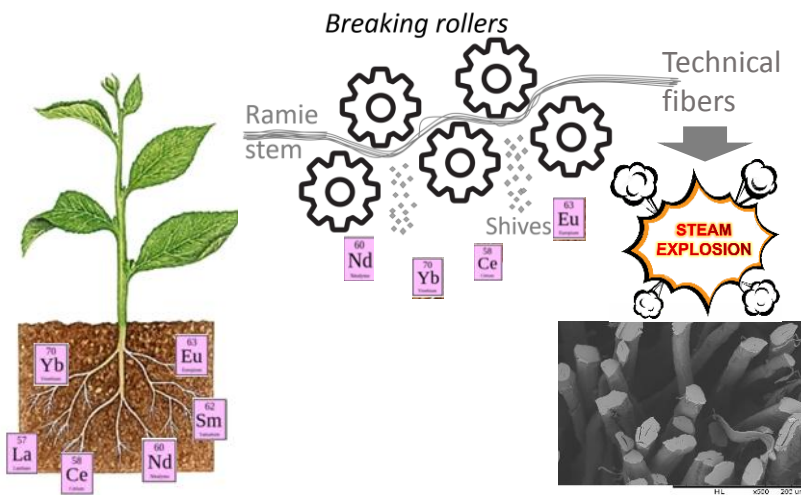
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Abstract : Ramie plants (*Boehmeria nivea*) have been cultivated on ion-adsorption rare earth elements (REEs) mine tailings and showed a low ability to accumulate REEs, especially in the stems ($\Sigma\text{REE} = 24 \pm 17 \text{ mg kg}^{-1}$) with higher concentration in leaves and roots ($\Sigma\text{REE} = 57$ to 66 mg kg^{-1}). Ramie individual fibers were isolated using three degumming processes: a traditional alkali treatment (AT), a steam explosion (SE) treatment after a neutral (SEN) or after a basic (SEB) impregnation. Cellulose-rich (> 80%) fine fibers with very low amounts of non-cellulosic sugars (<5%), low REE concentrations ($\Sigma\text{REE} \approx 4\text{-}6 \text{ mg kg}^{-1}$) and a high mechanical strength (tensile strength $\approx 260\text{-}540 \text{ cN/fiber}$, breaking point $\approx 20\text{-}50 \text{ cN/fiber}$) have been produced. However, SE degumming resulted in more degraded fibers with cracks slightly affecting their tensile properties.

Key words : Natural fiber, ramie, degumming, steam explosion, rare earth elements, phytoremediation

38
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40 **Graphical abstract :**
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57 **Statement of novelty:**

58 Ramie plant produced from a reclaimed rare earth elements rich mine tailing has been
59 evaluated for phytoremediation. The ramie fibers have been extracted using steam explosion a
60 green degumming technology.
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63

64 **Introduction**

65 In recent years, natural bast fibers (such as hemp, flax, jute, ramie) have received an
66 increasing attention due to their economic importance and their renewable and sustainable
67 character [1]. The main advantages of using natural fibers include **local availability**, low
68 density, biodegradability and carbon sequestration. Bast fibers are composed of elementary
69 fibers which are glued together by a cementing material composed of lignin and
70 polysaccharides [2]. Regardless of the final application concerned (textile or composite), the
71 removal of these non-cellulosic components (lignin, hemicelluloses, pectins) is required prior
72 to downstream processes through a degumming process. In fact, the gummy components
73 negatively affect the spinability of the fibers and, in composite applications, result in poor
74 strength properties of resulting materials. Typically, chemical methods are used for the
75 degumming process using large quantities of sodium hydroxide and other hazardous
76 chemicals [3].

77 Steam explosion (SE) is an efficient environment-friendly process for bast fibers degumming.
78 In a previous paper, we described the optimization of fine hemp fibers extraction using SE
79 [4]. This method allowed an efficient degumming of the technical hemp fibers and the
80 production of a cotton-like material. Recently, Jiang et al. [5] reported a green degumming
81 process of ramie using steam explosion in comparison with traditional chemical methods. It
82 has been concluded from this study that SE has a great potential for industrial developments
83 for economic and environmental reasons.

84 Compared to other natural fibers like flax or hemp fibers, ramie (*Boehmeria nivea*) fibers
85 display outstanding properties such as silk-like appearance, high mechanical strength and high
86 moisture absorption capacity and are among the most popular raw materials for textile
87 application [5-7]. Ramie is grown almost exclusively in China and one of the major limitation

88 of the large scale development of ramie industry is the utilization of arable land for the
89 production of non-food biomass. The utilization of plants for the management of polluted land
90 has been described in order to limit the dissemination of pollutants (phytostabilisation). In
91 addition, the cultivation of economic plants, such as fiber or bioenergy crops, can be
92 considered to contribute to the socio-economic development of contaminated areas [8,9].
93 Ramie, characterized by a vigorous root system, a high biomass and a good tolerance to heavy
94 metals, has been shown to be a good candidate for the phytostabilisation of contaminated soils
95 [10].

96 The demand for rare earth elements (REEs) is increasing worldwide due to their importance
97 in many high-tech products. In Southern China, the ion-adsorption type REE deposits are a
98 strategic resource. For decades, these deposits have been exploited by heap leaching, which
99 consists of the removal of the vegetation, the excavation of the soil layer enriched in REEs
100 and subsequent leaching by ammonium sulfate to recover REEs by ionic exchange. This
101 mining activity has generated important environmental damages, such as large areas of mine
102 tailings subjected to erosion, water contamination by ammonium, sulfate and metals, and
103 landslides. For instance, in Ganzhou city, Jiangxi province, mining activity to recover REEs
104 since 30 years has left more than 300 abandoned mines, 190 million tons of tailings and
105 destroyed more than 150 km² of forest [11]. Investigation of watersheds impacted by this
106 mining activity in the Dingnan county of Ganzhou city showed the contamination of river
107 waters and sediments and agricultural soils irrigated with contaminated water [12].

108 Main challenges for the revegetation of these tailings are the strong erosion and unfavorable
109 physical properties (sandy material, poor structure), the low levels of organic matter and
110 nutrients and the relatively high residual REE concentrations [13]. Phytoremediation using
111 amendments and pioneer or tolerant plants have been showed to improve the physico-

112 chemical properties of the tailings and to increase biological activity and diversity, suggesting
113 the potential of phytoremediation for the restoration of such mining areas [14,15].

114 In this context, the utilization of mining areas for growing industrial ramie could be a
115 promising approach not only for the revegetation and phytostabilization of ion-adsorption
116 REE mine tailings but also for biomass production. As a result, the mobilization of non-food
117 phytotechnology ramie fibers for industrial developments could be of particular interest in
118 order to reduce the impacts on the food markets.

119 In a previous work, we evaluated SE to achieve simultaneously the pretreatment and the
120 decontamination of Zn and Mn contaminated willow harvested from an experimental
121 phytoremediation plot for bioethanol production [16]. In the present study, we described the
122 defibration by SE and characterization of ramie fibers harvested on an ion-adsorption REE
123 mining area in Dingnan county (Jiangxi province) in China. REE concentrations in the
124 different compartment of the plant have been examined. The impact of the degumming
125 process used (SE or traditional chemical method) on the ramie fiber quality in terms of
126 chemical and mechanical properties has been examined.

127

128 **Materials and Methods**

129 **Raw material**

130 The site of demonstration for the reclamation of ion-adsorption REE mine tailings is located
131 in the Dingnan county of Ganzhou city, Jiangxi province. Details regarding the location,
132 reclamation program and growing conditions are given in the Supplementary Material section
133 S1. Small plants (1 year old, about 35 cm in height) of the ramie cultivar Zhongzhu No.1 were
134 planted in rows in the amended tailings with a density of 3 to 5 plants per m² in 2015. In 2018,
135 ramie stems (1-2 meters high, ~1 cm in diameter at the base) were sampled for the analysis of
136 fibers, cut and air-dried for several days.

137 **Carding and degumming**

138 Technical fibers were extracted from the ramie stems using a carding machine Bonino, type
139 250 HL520, with a lining adapted to bast fibers.

140 *Chemical degumming:* 10 g of technical fibers in 200 mL of NaOH 1% solution in water were
141 heated at 90°C with string during 1 h. The treated fibers were filtrated, washed with a dilute
142 acetic acid solution and then water.

143 *SE degumming:* Technical fibers were impregnated with a solution of water or of sodium
144 hydroxide (2 wt %). The fibers were macerated during 15 hours at room temperature using a
145 weight ratio water/biomass of 15/1 after what the biomass was filter-pressed (6.8 atm = 100
146 psi) to remove the excess water. The fibers were transferred into a 2 L steam gun where about
147 100 g (dry basis) of biomass was cooked at temperatures ranging from 170°C for 3 minutes
148 by addition of steam in a pressure-resistant reactor. After the cooking period, a pneumatic
149 valve was opened and the vapor phase exited the reactor through a nozzle entraining as well
150 the fibers.

151 The treated fibers were opened in the carding machine previously described (see below).

152 Opening the tufts into individual fibers: Card opened the tufts into the stage of individual
153 fibers. This is essential to enable the elimination of impurities and performance of the other
154 operations to be achieved as elimination of dust and removal of short fibers. Mainly for this
155 study to see the effects of the treatment on the opening of the fibers. The schematic of the
156 whole process is given in Fig. 1.

157 **Figure 1.**

158 **Chemical analysis**

159 The experimental details regarding Klason lignin and simple sugars % determination have
160 been given in previous papers. In short, extractive free fibers were ground and hydrolyzed in
161 2-steps procedure with sulfuric acid (72% and 4%). The insoluble residue (Klason lignin) is

162 recovered and weighted. The monosaccharide contents were determined from the liquid
163 fraction by HAPE-PAD (ICS-3000 Dionex) equipped with a Dionex CarboPac™ PA-20 (3 x
164 150 mm) analytical column. For experimental details see Simangunsong et al., 2018. The
165 stems, wood and fibers after treatment were analysed for trace elements at the Service d'
166 Analyse des Roches et des Minéraux (CNRS, Vandoeuvre-lès-Nancy, France). Trace element
167 concentrations were analysed using ICP-MS after lithium metaborate attack and acid
168 digestion [17]. Details regarding the soil analysis are given in Supplementary Material section
169 S2.

170 **SEM analysis**

171 Scanning electron microscopy (SEM) analysis was carried out with Hitachi 3000 microscope.
172 All images were taken at an accelerating voltage of 15 kV.

173 **Mechanical properties**

174 For measuring single fiber properties, Favimat (Textechno Herbert Stein GmbH and Co. KG,
175 Möchengladbach, Germany) was used. The main principle is that both single fiber ends are
176 clamped between two sets of jaws. The displacement is insured by a constant speed motor
177 with interchangeable equipment to vary the rate of elongation. For the tensile tests, the data of
178 the load and elongation were transferred to a computer to be plotted and analyzed. The
179 percentage of elongation and breaking point were determined according to the procedure
180 described in NF EN ISO 5079 1996 standard method (Textiles fibres – Determination of
181 breaking force and elongation at break of individual fibres). The tensile strength was
182 determined according NF T25-501-2 2015 (Reinforcement fibres – Flax fibres for plastics
183 composites-Part2: Determination of tensile properties of elementary fibres) standard method.

184

185 **SEM analysis**

186 Scanning electron microscopy (SEM) analysis was carried out with Hitachi 3000 microscope.

187 All images were taken at an accelerating voltage of 15 kV. Prior to the analysis, the
188 specimens were coated with a thin layer gold/palladium alloy using a sputter coater Quorum
189 SC7620

190 **Results and discussion**

191 Technical fibers were extracted in 32% yield from the ramie stems and separated from the
192 wood using a mechanical decortication process (see section 2). Three degumming methods
193 have been then compared: (1) a traditional alkali treatment, a SE treatment after (2) a neutral
194 or (3) after a basic impregnation (Fig. 1). The experimental conditions used for the
195 degumming steps were based on our previous studies using hemp technical fibers [4].

196 The compositions of ramie stem, wood, technical fibers and degummed fibers are given in
197 Table 1. Ramie stems displayed relatively high glucans (primarily from cellulose), lignin and
198 xylans contents. After decortication, the woody part retained most of the lignin and xylans,
199 the fibers recovered being cellulose-rich and relatively poor in lignin (< 10%). Glucomannan
200 has been identified as one of the hemicelluloses of ramie [18]. From the mannose content in
201 the different compartment of the plant listed in Table 1, we can assume that glucomannan is
202 closely associated to the cellulosic fibers whereas xylan is one of the main components of
203 wood. GluA, a marker of pectic components, was recovered in the fiber fraction as well. From
204 Table 1 and in accordance with previous works dealing with ramie fibers degumming, it can
205 be seen that the steam explosion with a basic impregnation is an efficient degumming process,
206 producing cellulose-rich fibers ($\approx 85\%$) with very low amounts of non-cellulosic sugars
207 (<2%). In our study, the relatively high residual lignin contents obtained are justified by the
208 degumming conditions applied, without bleaching agent.

209 The composition of the stems and extracted wood, technical fibers and fine fibers in terms of
210 Trace Elements (TE) including Rare Earth Elements (REEs) is given in Table 1. The
211 distribution of trace elements in the soil and organs of ramie grown on amended tailings is

212 reported in Supplementary material section S2. In the plants, the average REE concentrations
213 were higher in leaves and roots than in the stems. The major REEs detected in the plant were
214 La, Ce and Nd as well as Y; the other REEs were present in low concentrations ($<1 \text{ mg.kg}^{-1}$).
215 The REE bioconcentration factor (leaf concentration/soil concentration) was 0.12 ± 0.07 and
216 the REE translocation factor (leaf concentration/root concentration) was 1.3 ± 0.7 , attesting that
217 ramie has relatively poor ability to accumulate REEs. Main other trace elements in the plant
218 were Ba, Sr, Rb and Zn (see Supplementary material section S2).

219 **Table 1**

220 After degumming, it can be observed a strong decrease of REE concentrations in the fine
221 fibers (Table 1), which could be explained by the removal of a part of the pectic
222 polysaccharides which are known to exhibit properties of ions exchange with high affinity for
223 metal ions.

224 As previously proposed, in the SE process the combination of the steaming step (which
225 provoked the hydrolysis of non-cellulosic components) and the shearing forces (applied
226 during the explosive decompression) increased the extraction ability of TE in the aqueous
227 stream [16]. As a result, the degumming step appears to be an efficient process for both (1)
228 cleaning TE-enriched phytoremediation-borne ramie fibers and (2) producing superior quality
229 fine fibers with low variability.

230 Degumming processes produced isolated ramie single fibers having an average diameter of 30
231 (± 5) μm as can be seen in the SEM images given in Fig. 2. In contrast with soda treatment
232 which produced smooth and intact fibers (Fig. 2A), SE degumming resulted in more degraded
233 fibers with cracks and a rough surface (Fig. 2B and C). This can be explained by a
234 degradation under the harsh SE conditions used as previously observed for hemp fibers [4].

235 The tensile properties of the fine ramie fibers produced were also analyzed in terms of
236 elongation, breaking point and tensile strength. All the results obtained are gathered in Table

237 2. The data for cotton fiber was also used as a reference in order to evaluate the ramie fibers
238 produced in this work in textile area. The results obtained compared well with literature:
239 fibers extensional strain at break is lower for ramie ($\approx 4\%$) than for cotton fibers ($\approx 9\%$) but
240 regarding Breaking Point and Tensile Strength (in cN), ramie fibers displayed higher
241 properties [2, 5, 7]. The Elongation and Breaking point of ramie fibers produced by a
242 chemical degumming process ($\approx 4.5\%$ and ≈ 47 cN respectively) displayed significantly
243 higher values than exploded fibers ($\approx 3.4-3.9\%$ and $\approx 22-30$ cN respectively). These results
244 can be related with the aspect of the fibers given in the Fig. 2, the presence of damaged fibers
245 after SE degumming having probably a negative impact on the tensile properties.

246

247 **Fig. 2.**

248

249 **Conclusion**

250 In terms of phytomanagement, ramie can be cultivated on ion-adsorption REE mine tailings
251 and showed a low ability to accumulate REEs, especially in the stems and in the fibers. From
252 this study we can conclude that SE appeared to be an efficient and green process for both
253 degumming and cleaning phytotechnology ramie fibers. However, the relatively harsh
254 conditions used during the SE degumming step resulted in more degraded fibers slightly
255 affecting their tensile properties. This first field trial on REE ion-adsorption mine tailings
256 showed the feasibility of producing industrial ramie fibers from plants growing on such
257 substrate but further work is needed to optimize the crop management, the degumming step
258 and the utilization of the woody part for a sustainable whole valorization of the ramie plant.

259

260 **Acknowledgement**

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264

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336 **Table 1. Chemical composition of ramie stems, and extracted wood and fibers and distribution of**
 337 **trace elements including Rare earth elements (REEs) in the organs of ramie plants.**
 338

	<i>Chemical composition</i>					<i>Trace elements</i>		
	K Lignin % ⁴	Glc ⁵	Xyl ⁶	GalU ⁷	Man ⁸	Rb	Zn	ΣREE ⁹
whole stems	19.7±1.4	48.9±1.2	9.3±0.3	2.7±0.1	1.5±0.1	53±1.9	70±2.1	17.4±0.4
wood	21.4±1.2	51.8±0.9	13.3±0.7	3.2±0.2	0.3±0.0	49±1.1	60±1.8	14.9±0.3
Raw fibers	9.7±0.7	77.4±1.3	0.4±0.0	3.1±0.2	2.0±0.1	71±2.2	100±3.5	6.4±0.1
Fine fibers								
AT ¹	7.6±1.1	82.5±0.6	0.4±0.0	2.1±0.1	2.0±0.2	1±0.0	76±3.2	5.7±0.1
SEN ²	7.2±1.6	81.6±1.1	0.5±0.0	2.3±0.2	2.7±0.2	3±0.0	61±3.0	4.5±0.1
SEB ³	9.9±0.2	84.1±0.9	0.3±0.0	0.6±0.0	0.7±0.1	<1	53±2.7	4.4±0.1

339 ¹alkali treated fibers, ²steam exploded fibers after water impregnation, ³steam exploded fibers after basic
 340 impregnation, ⁴Klason lignin, ⁵Glucose, ⁶Xylose, ⁷galacturonic acid, ⁸mannose, other sugars (Ara, Rha, Gal and
 341 GlcU were also detected at low levels (< 1%) ⁹sum of the concentrations of the 14 lanthanides and Y.
 342
 343
 344

345 **Table 2. Physical properties of ramie fibers extracted with three different degumming processes,**
 346 **compared with cotton fiber.**

347			
<i>Mechanical properties</i>			
fibers	Elongation (%)	Breaking point (cN)	Tensile strength (cN/fibre)
AT ¹	4.6±4.5	47.1±25.7	259.1±210.1
SEN ²	3.9±3.2	30.3±12.9	537.8±692.2
SEB ³	3.4±1.6	22.1±13.3	438.7±619.4
cotton	9.4±5.9	3.9±1.9	82.7±76.7
cotton ¹⁹	9.7	5.6	-

353 ¹alkali treated fibers, ²steam exploded fibers after water impregnation, ³steam exploded fibers after
 354 basic impregnation

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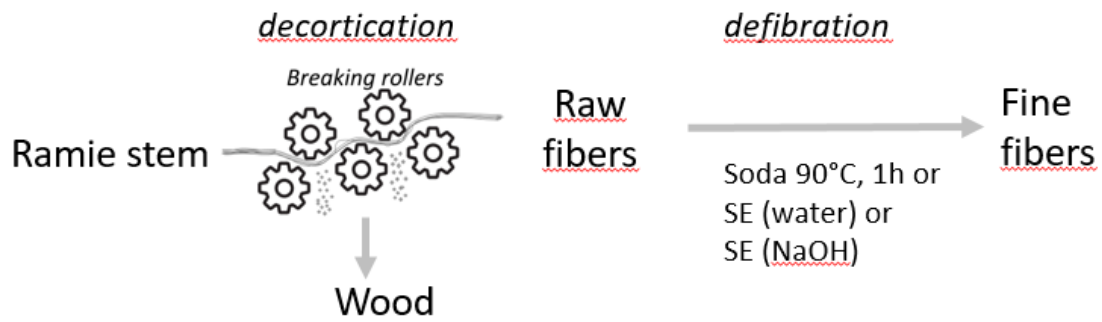
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366 **Figure 1. Schematic of the process.**

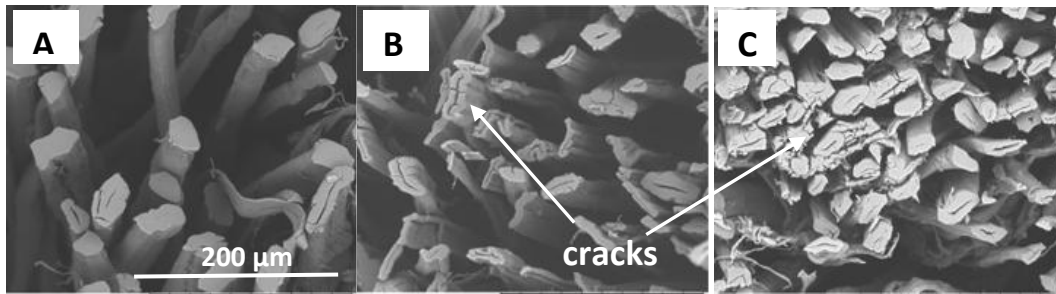
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371 **Fig. 2. Scanning Electron Microscopy images (x500) of Soda fibers (A), SE treated fibers after water**
372 **impregnation (B) and SE treated fibers after soda impregnation (C).**



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SUPPLEMENTARY MATERIAL

383

**Industrial Ramie growing on reclaimed ion-adsorption Rare Earth
384 Elements mine tailings in Southern China: Defibrillation and fibers
385 quality**

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SI- Reclamation Program and growing conditions

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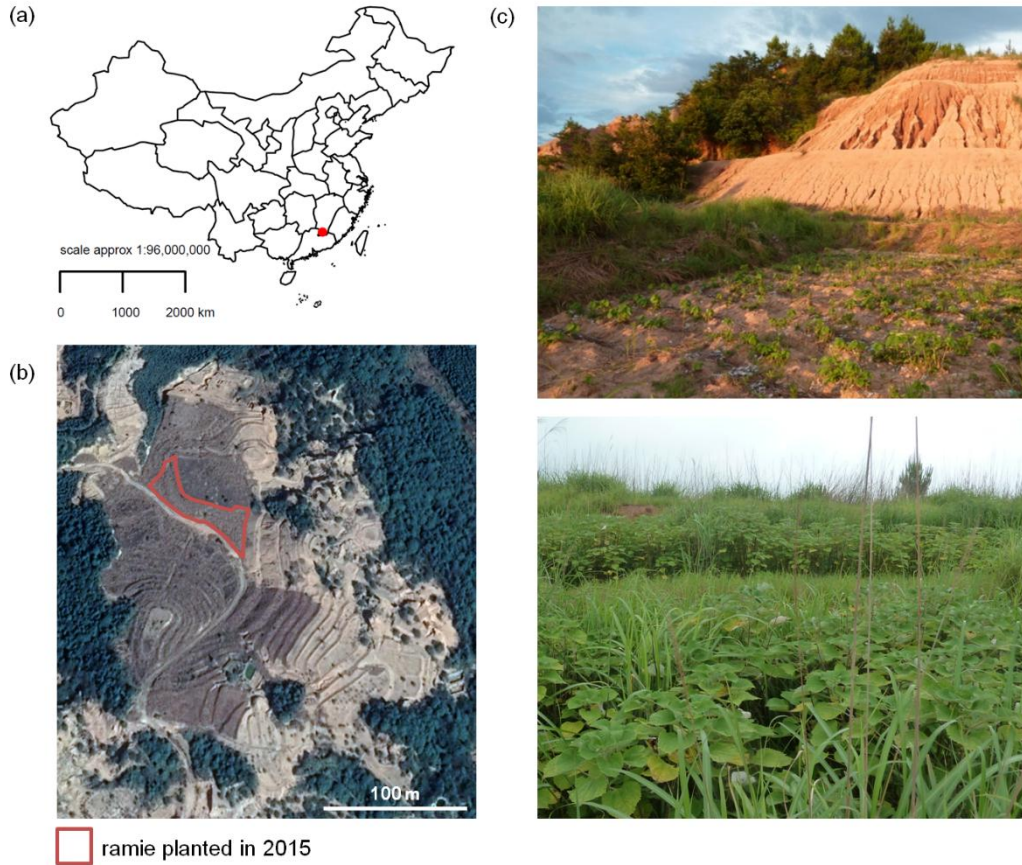
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The site is located in the Dingnan county of Ganzhou city, Jiangxi province (latitude:
24.989070761774737°, longitude: 115.0527710298679°). The climate is subtropical monsoon
humid, with an average annual temperature of 18.8°C and average annual rainfall of 1609 mm
(Dingnan County People's Government Network). Natural soils of the area are red soils,
formed by the weathering of granitic rocks under subtropical climate. Abandoned since
around 10 years, the mine tailings (53 ha) were landscaped in terraces, with steep slopes (>
45°C) subjected to strong erosion in some parts of the site. The vegetation cover was very
limited and composed mainly of pioneer species (e.g. *Miscanthus sinensis*, *Pinus
massoniana*). Since 2015, a reclamation program has been implemented using different plants

411 (grasses, fiber plants and hyperaccumulating plants), including ramie (*Boehmeria nivea* (L.)
 412 Gaudich).



413

414 **Figure S1- Cultivation of ramie on reclaimed ion-adsorption REE mine tailings**

415 (a) localization of the site in the Dingnan county of Ganzhou city, Jiangxi province, China (b) aerial
 416 view of the site taken in 2018 showing the reclamation program with grasses and fiber plants with
 417 the delimitation of the plots planted with ramie since 2015 (source: Google Earth) (c) pictures of the
 418 ramie cultivated on the mine tailings taken in 2016

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420 Ramie was planted on several terraces (1250 m² in total) in July 2015. Fresh organic
 421 amendment was added with a rate of 50-60 t ha⁻¹ and mixed within the first 12 cm of the
 422 tailings by tillage. One-year plants (about 35 cm in height) of the ramie cultivar Zhongzhu
 423 No.1 were brought from the Institute of Bast Fiber Crops (Chinese Academy of Agricultural
 424 Sciences) and planted in rows in the amended tailings with a density of 3-5 plants per m². In
 425 June 2016, the aboveground part of the plants was cut and a granulated NPK fertilizer was

426 added with a rate of 450 kg ha⁻¹. In March 2018, invading plants and aboveground part of
 427 ramie were cut and amendment was added to improve the growth of ramie plants. Plants and
 428 soil associated to the
 429 root system were sampled in May (5 samples), July (3 samples) and November 2016 (3
 430 samples) at different locations of the planted terraces. Soil associated to roots was collected
 431 by shaking, air-dried and sieved at 2 mm. All the plants were divided into roots, stems and
 432 leaves and washed with tap water using ultrasound bath then rinsed with deionized water.
 433 Then they were dried at 60°C and ground. In November 2018, the stems of several plants
 434 were sampled for the analysis of fibers. Some stems reached 2 m in height. The stems were
 435 cut and air-dried for several days.

436

437 ***S2- Distribution of trace elements in the soil and plant samples collected in 2016 (mean±standard***
 438 ***deviation, n=11)***

439

	Ba	Rb	Sr	Zn	REEs (in mg kg ⁻¹)				Sum ¹
					La	Ce	Nd	Y	
Leaves	249±128	50±33	59±24	260±74	17.2±6.1	7.1±6.6	8.7±3.8	23±12	57±36
Stems	155±157	63±31	16±4	149±57	10.7±6.6	3.0±1.5	4.1±1.8	6.3±3.4	24±17
Roots	82±56	38±19	11±3	118±36	19±15	7.8±2.8	10.1±7.3	16±18	66±52
Soil	584±111	295±32	34±4	165±36	131±59	173±33	81±33	107±58	592±197

440 ¹sum of the concentrations of the 14 lanthanides and Y