

## “Functional upcycling” of polymer waste towards the design of new materials

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## **1. Polyethylene terephthalate (PET)**

### **1.1 Mechanical recycling of PET**

For PET as a thermoplastic, extrusion followed by casting is still the most applicable industrial process for mechanical recycling<sup>1, 2</sup>. However, every cycle of heating and mechanical treatment leads to sufficient deterioration of properties, which reduces the cost of the final product<sup>2, 3</sup>. The described issues can be partially solved by the simultaneous formation of composites with inorganic fillers or other polymers<sup>4, 5</sup> and the addition of special reagents preventing the undesired degradation of polymers backbone<sup>6-8</sup>.

Obviously, the traditional method for PET recycling has been reviewed in several recent papers<sup>9-12</sup>. Here, we are highlighting a few examples of rational waste management through the formation of PET-based composites. Waste PET was used to prepare super-thin fibrous scaffolds with improved mechanical characteristics by electrospinning<sup>13</sup>. The prepared filter devices are used for air filtration from condensable hydrocarbons and solid particles, in particular tobacco smoke. It is noteworthy that the use of the described method does not lead to structural changes in the PET polyester chain. Rangari *et al.* combined waste PET bottles and packages to produce biochar with improved mechanical and thermal performance<sup>14</sup>. Packages were pyrolyzed to biochar, while PET bottles were shredded for extruding into a 3D-printable filament, resulting in improved properties – tensile modulus, dynamic modulus, dimensional stability, and thermal stability in an oxidative environment<sup>14</sup>.

Despite the significant success in the field, the mechanical recycling of waste PET suffers from common drawbacks related to a decrease of polymer lifespan and the production of materials with lower cost. Taking into account the reactivity of PET, the advanced methods of chemical recycling or functional upcycling occupy a central stage in the PET utilisation for the circular economy.

### **1.2 Chemical re-/upcycling of PET**

Methods for the chemical processing of waste PET are characterized by either complete or partial depolymerization of the polymer chain<sup>15</sup>. Due to the chemical nature of PET, the C–O bond can be readily cleaved by a wide range of nucleophilic reagents with the formation of terephthalic acid (TA) and ethylene glycol monomers or their derivatives. For instance, two main approaches for the depolymerization of PET are well-established and connected with the interaction with O-centered nucleophiles – water (hydrolysis) or glycols (glycolysis)<sup>15-17</sup>. Nevertheless, reactions with

N-centered nucleophiles, such as amines or hydrazines, have been also applied for the preparation of functional derivatives of TA (amides, hydrazides, *etc.*)<sup>18-20</sup>.

It should be noted that the classic approach for the depolymerization of PET has been recently applied for the preparation of novel materials with exciting properties. For instance, *in situ* conversion of glycolized PET to higher-value fiber-reinforced plastics was suggested<sup>21</sup>. Beckham *et al.* reported preliminary decomposition of waste PET by glycolysis, followed by transesterification by malate or fumarate alcohols *via* melt blending. In the next step, olefin-containing carboxylic acid, either methacrylic or acrylic acids, were used to prepare acrylates by radical polymerization. Finally, cross-linking with Bondo fiberglass mat was applied to convert olefinic polymer to fiber-reinforced plastics<sup>21</sup>.

In turn, the recent work of Jason and Fam *et al.* demonstrated a fundamentally new sustainable approach to the production of polyurethane electrolytes from waste PET bottles, which consists in the depolymerization of a polyester PET chain by aminolysis with ethanolamine to form bis(2-hydroxyethyl)terephthalamide (BHETA) in the presence of an organocatalyst. In a subsequent step, the BHETA monomer was mixed with polyethylene glycol and reacted with hexamethylene diisocyanate to form a polyurethane containing hard (BHETA): and soft (polyethylene glycol) blocks<sup>22</sup>. Using PET-derived PU electrolytes as solid and gel polymer electrolytes, a working lithium-ion battery can be assembled, demonstrating promising ionic conductivity.

Among non-selective approaches to chemical recycling, thermal pyrolysis is still the most applicable<sup>23</sup>. Despite the complex composition of reaction products, the resulting mixtures have a considerable commercial value as industrial lubricants, additives to oils, *etc.*<sup>15</sup>

As a carbon-rich polymer, PET has been involved in the carbonization process for the formation of various carbon-based materials. In recent years, such process has been used for the production of porous materials for supercapacitors<sup>24</sup>, adsorbents for CF<sub>4</sub> removal<sup>25</sup>, CO<sub>2</sub> capture<sup>26</sup> or quantum dots preparation *via* preliminary PET aminolysis<sup>27</sup>. However, energy consumption for pyrolysis or carbonization is high due to increased reaction temperature (600-700 °C).

A technologically appealing alternative to traditional methods is photochemical valorization, which directly utilizes solar energy to transform plastic pollutants into chemicals and fuels at room temperature. In 2018, Reisner's group firstly reported photocatalytic H<sub>2</sub> evolution from PET bottles using semiconductor CdS/CdO<sub>x</sub> quantum dots, accompanied by the conversion of the polymer into organic products (formate, acetate and pyruvate)<sup>28</sup>. Later, more advanced photocatalytic systems have been proposed: C<sub>3</sub>N<sub>4</sub><sup>29</sup>, MoS<sub>2</sub>/Cd<sub>x</sub>Zn<sub>1-x</sub>S<sup>30</sup>, carbon nitride/nickel phosphide (CN<sub>x</sub>|Ni<sub>2</sub>P)<sup>31</sup>.

Even more, electrochemical recycling of PET has recently been demonstrated by several research groups. Duan *et al.* reported electrocatalytic PET upcycling to potassium diformate and TA together with H<sub>2</sub> production using a bifunctional CoNi<sub>0.25</sub>P electrocatalyst in KOH. Alternatively, the Cu-based electrocatalytic system was applied for selective electrochemical degradation of PET forming TA and formate<sup>32</sup>. A similar catalytic activity was demonstrated by cobalt-based spinel oxide (CuCo<sub>2</sub>O<sub>4</sub>) nanowire arrays on Ni-foam<sup>33</sup>.

The chemical re-/upcycling of PET was intensively reviewed previously in a range of publications<sup>17, 19, 34</sup>. Chemical methods for waste PET processing are well-investigated and could be used for the preparation of valuable chemicals. Nevertheless, the proposed approaches are mostly limited by degradation of C–O bonds in the polymer backbone.

## **2. Polyethylene (PE)**

### **2.1 Mechanical recycling of PE**

Mechanical recycling of PE imposes physical extruding and mixing of PE waste with other materials to design composites with improved characteristics<sup>9, 35</sup>. As an inexpensive additive, PE is mostly implemented as asphalt additive for pavements<sup>36, 37</sup>, rubber additives<sup>38</sup> and in wood plastic composites for various applications, which include building, interior decoration, door and window making, flooring, *etc.*<sup>39-41</sup> Recycled low-density PE (LDPE) and high-density PE (HDPE) could be implemented in lithium liquid lubricant grease<sup>42</sup>, internal layer in multi-layer food packaging<sup>43, 44</sup>. However, PE is often subjected to thermo- and photo-oxidative degradation during the processing or use, responsible for simultaneous chain scission and cross-linking of the polymer chains. Indeed, such undesired transformations lead to the sufficient deterioration of properties limiting the further application of the recycled polymers.

### **2.2 Chemical re-/upcycling of PE**

Chemical recycling of PE requires cleavage of inert and robust C–C bonds in the polymer backbone. However, this process is hindered by a prohibitive amount of required energy and low selectivity<sup>45</sup>. Chemical recycling has been reviewed as a separate chapter in recent reports<sup>46, 47</sup>. Here, we will briefly overview the main recent directions of chemical recycling and its applications.

Traditional thermal and catalytic pyrolysis are the most applied chemical strategies in the industry for PE recycling<sup>48, 49</sup>. Indeed, such processes necessitate the utilisation of heterogeneous catalysts bearing acidic active centers at elevated temperatures. The catalytic pyrolysis of PE is

usually unselective, resulting in a broad distribution of gas (C<sub>3</sub> and C<sub>4</sub> hydrocarbons), liquid (cycloparaffins, oligomers, and aromatics), and solid (char, coke) products<sup>50, 51</sup>.

The next common strategy is converting polyolefins to dicarboxylic acids using strong oxidants such as O<sub>2</sub>, NO, HNO<sub>3</sub>, H<sub>2</sub>O<sub>2</sub> at high temperatures or under deep UV-irradiation<sup>52, 53</sup>. The selectivity of the oxidation process as well as the distribution of acids largely depend on the reaction conditions, which limited the industrial application of such processes. Nevertheless, optimization of the reaction conditions allowed to develop novel approaches towards the selective oxidative cleavage of PE chains. Thus, sufficient upgrade has been achieved by implementation of Fenton reagents as oxidants (main products – butanedioic, acetic acids)<sup>54</sup>, or alternative approaches towards reaction activation, such as microwave-assisted method (main products – succinic, glutaric, and adipic acids)<sup>55</sup> and photocatalytic oxidation (products – acetic and formic acids)<sup>56, 57</sup>.

Recently, the focus of attention has been directed toward hydrogenolysis of PE under catalytic conditions<sup>45, 58</sup>. Despite the efficiency of the process, it requires the addition of complicated catalytic systems based on Pd, Pt, and Ru. Another exciting approach is connected with a tandem catalytic cross-alkane metathesis (CAM) in the presence of iridium catalysts, leading to waxes and liquid fuels as recycled products<sup>45, 59, 60</sup>. Despite the progress in reductive hydrogen-involved transformation, expensive and demanding catalysts are required.

Obviously, PE, as a rich carbon source, has been involved in the carbonization process with the formation of various high-value carbon materials<sup>61, 62</sup> for application as energy storage electrode materials in batteries<sup>63-66</sup>, and supercapacitors<sup>67, 68</sup>, organic photovoltaic cells<sup>69</sup>, sensors<sup>70, 71</sup>, adsorbents for oil-water separation<sup>72</sup>, and capture of metal cations<sup>73</sup>.

### **3. Polyvinyl chloride (PVC)**

#### **3.1. Mechanical recycling of PVC**

Mechanical recycling can be applied to PVC in case of purified waste using a proper stabilizing agent to neutralize evolving HCl<sup>74</sup>. Nevertheless, adding stabilizers on each recycling step impacts negatively the final product's performance and requires toxic plasticizers<sup>9, 75</sup>. The alternative pathway is mixing crushed PVC with other materials to generate composites with improved performance in building and construction<sup>76-80</sup>, conveyer belt<sup>81</sup>, and insulation<sup>82</sup>. It should also be noted that there are a few examples of mechanical recycling with the formation of smart materials – sorbents<sup>83, 84</sup> or antimicrobial composites<sup>85</sup>.

Despite the simplicity of mechanical recycling, PVC plastics are insufficiently suitable for the widespread application of recycled products. This is due to the intrinsic properties of PVC,

such as relatively low thermal and photostabilities<sup>9</sup>. Moreover, the high risk of HCl evolution under usage limited the wide application of PVC-based composites.

### **3.2. Chemical re-/upcycling of PVC**

Due to the disadvantage of mechanical methods, chemical recycling becomes essential for the PVC waste management. The common way for the cleavage of PVC polymer backbone is depolymerization, cracking and gasification<sup>86, 87</sup>. Indeed, the presence of chlorine leaves a stamp on the chemical processes due to the possibility of the generation of harmful organics (*e.g.*, dioxins) and hydrochloric acid. Thus, PVC chemical recycling always starts with dehydrochlorination in dry or wet conditions, preventing poisoning effect and corrosion of reactors<sup>88-92</sup>. Dry methods rely on simple heating in presence of different additives (alkalines, Ni<sup>2+</sup> or Cu<sup>2+</sup> ions, and others)<sup>93-95</sup>. Wet processing comprises bimolecular elimination or nucleophilic substitution in polar solvents, such as *N,N*-dimethylformamide, tetrahydrofuran (THF), dimethyl sulfoxide, ethylene glycol, *etc.*, leading to the C–Cl bond cleavage. Obviously, the wet approach accomplishes a higher dechlorination degree under milder conditions, but requires expensive solvents and complicated regeneration procedures.

Wet and dry processes can lead to the transformation of PVC to syn-gas (CO+H<sub>2</sub>) and mixtures of hydrocarbons<sup>88, 96</sup>, benzenes<sup>84, 97</sup>, polycyclic aromatic hydrocarbons<sup>97</sup>, char<sup>84</sup>, organic acids<sup>94, 98</sup>, and fuels<sup>56, 99</sup>. Besides that, the number of studies describing PVC carbonization to sp<sup>2</sup> carbon and the application of the produced materials as electrode in supercapacitors<sup>100, 101</sup>, lithium-ion batteries<sup>102</sup> and graphene preparation<sup>103</sup> is growing up. One of the most recent examples is using CO<sub>2</sub> to convert waste PVC into formate at 300 °C, accompanied by PVC dechlorination<sup>104</sup>.

Chemical recycling of PVC wastes has become one of the most intensively developing fields of research (the detailed reviews<sup>105, 106</sup>). However, the most explored approaches require high temperatures, complicated catalytic systems, and complex equipment, which substantially increase the costs of the process<sup>48, 88</sup>. Thus, the development of recycling processes of PVC wastes with the formation of products with added value has a bright perspective on the circular economy.

## **4. Polypropylene (PP)**

### **4.1. Mechanical recycling of PP**

Mechanical recycling of PP imposes the physical mixing of PP waste with other materials to design composites with improved characteristics through melting/extraction/pumping/blending. As an inexpensive additive, PP is mostly implemented in asphalt for pavements<sup>107, 108</sup>, concrete<sup>109</sup>,

<sup>110</sup> and in wood-plastic composites<sup>111</sup>. The PP fibers waste has been used as an insulator in buildings<sup>112</sup> or as a reinforcement material in sandy soil.

Moreover, mixing of PP waste with different fillers (loofah sponge, inorganic fillers, wood sawdust, ground sunflower husk or other polymers) was reported for mechanical properties enhancement<sup>113-117</sup> including for 3D-printing<sup>118</sup>. The application of mechanically recycled PP is not limited to construction, PP surgical face masks, prepared from waste, showed a similar performance to that of commercial ones<sup>119</sup>. Mechanical recycling has been reviewed as a separate chapter in a recent article<sup>9</sup>.

One of the non-common methods for the purification and reutilisation of used PP masks was developed recently. This approach is based on the dissolution-precipitation of polymers using green solvents (*p*-cymene and ethanol)<sup>120</sup>. Such treatment allows to remove additives, impurities, and bio-contaminants, as well as to prepare fine powders for further membrane preparation<sup>120</sup>. The same approach was implemented for upcycling PP (face masks and sack) waste to emulsion templated, sacrificial 3D-printed porous funnel and to PE-coated mesh for efficient oil-water separation<sup>121, 122</sup>.

## 4.2. Chemical re-/upcycling of PP

PP is an inert polymer with enhanced thermal stability, making its chemical recycling very challenging. The chemical catalytic recycling of PP to monomers is hindered by a prohibitive amount of required energy and low process selectivity. Due to this reason, modern technologies for recycling are concentrated on the preparation of liquid and gaseous fuels and chemicals<sup>123</sup>.

A more prospective reaction for PP recycling is hydrogenolysis catalyzed by Ru at 200-250 °C and 20-50 bar H<sub>2</sub><sup>124</sup>, resulting in high-value liquid alkanes and alkylarenes production used as feedstocks to produce surfactants, lubricants, refrigeration fluids, and insulating oils<sup>124-126</sup>.

The opposite approach is based on the catalytic oxidation of polyolefins to dicarboxylic acids at elevated temperatures (100-150 °C) in O<sub>2</sub> atmosphere<sup>127</sup>. Later, milder methods, such as Fenton reaction<sup>128</sup> or microwave plasma treatment<sup>129</sup>, have been introduced for the oxidative transformations of PP.

However, traditional thermal or catalytic (metal oxides, zeolites, and silica-alumina catalysts) pyrolysis of carbon-rich PP is the most promising technology of waste disposal for energy recovery<sup>49, 130</sup>. PP pyrolysis produces a series of high-value products, including syngas<sup>131, 132</sup>, C<sub>1</sub>-C<sub>4</sub> hydrocarbons<sup>132-134</sup>, organic liquids (cycloparaffins, oligomers, and aromatics)<sup>133</sup>, bio-oil<sup>135</sup>, and solid products (char, coke)<sup>133, 136, 137</sup>. The pyrolysis conditions pre-determine the properties of possible products and their applications ranging from energy storage in sodium-ion

batteries<sup>138</sup>, lithium-sulfur batteries<sup>139, 140</sup>, supercapacitors<sup>141</sup>, electrocatalysts<sup>142</sup> to microwave absorbers<sup>143</sup>, CO<sub>2</sub>-capture materials<sup>144</sup>, dyes adsorption<sup>133, 145</sup>, and adsorbents for metal cations or oils<sup>131, 145</sup>. Chemical recycling has been reviewed as a separate chapter in recent publications<sup>46, 47</sup>.

## **5. Polystyrene (PS)**

### **5.1 Mechanical recycling of PS**

Mechanical recycling of PS imposes the common physical mixing of grounded PS with other materials for the preparation of composites with improved characteristics<sup>9</sup>. Mostly, this strategy was applied in the field of construction materials, where PS serves as the typical filler for the improvement of performance and technological properties of material<sup>146, 147</sup>. The most common matrixes are wood<sup>148</sup>, cement<sup>149</sup>, sand, wire mesh, concrete mixture and light-weight brick<sup>147</sup>. Nevertheless, PS generally has poor strain resistance, low strength and high inflammability, which limit its usage in the construction and building<sup>150</sup>.

### **5.2 Chemical re-/upcycling of PS**

Similar to other polymers, chemical recycling of PS requires cleavage of unreactive C–C bonds in the polymer backbone, leading to small molecules, such as monomer, fuels<sup>151</sup> and chemical feedstocks<sup>152</sup>, which requires a sufficient energy consumption. For instance, PS thermal depolymerization necessitates temperatures up to 500 °C<sup>153</sup>.

While pyrolysis of PS into styrene monomers is feasible<sup>154</sup>, this plastic can also be a valuable source of small, oxygenated molecules, which cannot be obtained through traditional pyrolysis. The most common chemical strategies to recycle PS are oxidative cleavage using oxidizing gases (O<sub>2</sub> or NO for the production of benzoic acid derivatives)<sup>52, 155, 156</sup>, pyrolysis in reductive/inert conditions at T > 300 °C (main products are benzene, toluene)<sup>157</sup>, Fenton reaction, including photo-induced (products are sulfonic acids)<sup>158</sup>. Apart from preparation of small molecules from PS, the controllable carbonization of PS with the full breakage of C–C bonds leads to the formation of sp<sup>2</sup>-hybridized carbon materials (nanotubes, spheres, porous sheets). PS can be recycled into pre-designed microstructures to prepare carbon-based electrodes for supercapacitors<sup>159, 160</sup> and lithium-ion batteries, catalyst supports, solar steam evaporators<sup>146, 161</sup>. The chemical recycling has been reviewed as a separate chapter in recent reviews<sup>156, 162, 163</sup>.

However, due to the presence of phenyl groups and single C–C bonds, the decomposition or depolymerization process of PS into its monomers is an energy-intensive process requiring high temperatures and pressure. Moreover, toxic and carcinogenic polyaromatic hydrocarbons could be



formed during the high-temperature treatment. From an economic point of view, chemical recycling is not suitable for petroleum-based plastics, because petrochemical feedstocks are cheaper than recycling products<sup>150</sup>. Therefore, the main challenge is to find an alternative and balancing strategy for PS recycling.

## **6. Polyurethane (PU)**

### **6.1. Mechanical recycling of PU**

Mechanical recycling of PU wastes is proceeded by regrinding, rebinding, adhesive pressing, injection or compression molding<sup>164</sup>. The produced powder with various granulometric compositions, prepared by grinding, cutting, or tearing, can be used as fillers in newly manufactured PUs<sup>165</sup>. However, this approach suffers from relatively low quality of resulting material and elevated expenses of the processing. Waste PU from bumpers can be condensed under high pressure and temperature (180 °C, 350 bar) by hot compression molding to create PU with a more rigid structure. Alternatively, adhesives, which consist of a PU compound, can be mixed with scraps or pellets to generate high-density PU for floor coverings and sports matting due to their durability and flexibility<sup>166</sup>. Recently, mechanical recycled PU is applied for construction and padding-type materials<sup>165, 167, 168</sup>. PU can be considered as one of the most recycled polymers by mechanical treatment. Nevertheless, the additional grinding of PU leads to the formation of increased amount of microplastic<sup>169</sup>, which can be considered as the most hazardous among other polymers<sup>169, 170</sup>.

### **6.2. Chemical re-/upcycling of PU**

PU is the product of polycondensation between diols (or polyols) with diisocyanates or polyisocyanates with the formation of carbamate moieties. The chemical nature of such groups makes favorable a wide range of reactions, leading to the cleavage of C–O or N–C bonds with the formation of merit chemicals. Similar to PET, the most well-studied approaches are hydrolysis, glycolysis, and aminolysis<sup>171</sup>, which are based on the reactivity of carbamate groups and ability to interact with nucleophiles.

Hydrolysis and hydroglycolysis involve interaction with water in the presence of lithium hydroxide as a catalyst at a temperature of 200 °C<sup>172</sup>. The other approach, glycolysis, is relative to hydrolysis, but, in this case, alcohols serve as nucleophiles in the transesterification, which allow the recovery of the polyols for the next cycle of PU production<sup>173</sup>. Aminolysis concerns the interaction with amines, leading to isolation of bi- or polyfunctional amines and alcohols<sup>174</sup>. PU

also can undergo phosphorolysis using phosphoric and phosphonic acids. Such process allows the preparation of a mixture of phosphorus-containing oligo-urethanes used as flame retardant additives in novel PU-based materials<sup>175</sup>.

PU can be processed *via* gasification approach with the formation of syngas directly in the blast furnace<sup>176, 177</sup>. Nevertheless, the common pyrolysis procedures with the formation of carbon-based materials are limited by the evaporation of toxic compounds such as hydrogen cyanide, benzene, or aniline<sup>178</sup>. However, some recent examples demonstrated the preparation of adsorbents for H<sub>2</sub>S and CO<sub>2</sub> by carbonization of PU in inert or hydrothermal conditions at temperatures of 160-400 °C<sup>179, 180</sup>.

Unfortunately, the chemical or thermochemical recycling of PU is limited by the release of toxic compounds, and high energy input either to heat up the batch or to apply the required pressure. The most recent overview of PU chemical recycling is discussed in<sup>164, 181</sup>. Due to this reason, the methods of functional upcycling become more important for the prolongation of PU lifespan without sufficient loss of properties.

## **7. Poly(methyl methacrylate) (PMMA)**

### **7.1. Mechanical recycling of PMMA**

Mechanical recycling is one of the simplest methods distinguished by relatively low carbon footprint, but requiring complicated procedures for the separation from other polymers<sup>182, 183</sup>. Mechanically recycled PMMA is limited in common optical applications, because of the deterioration of the optical properties during crushing, powdering, and even dissolution<sup>184, 185</sup>. Nevertheless, the recycling of PMMA from liquid-crystal display (LCD) monitors<sup>186</sup> with the extrusion process into polymer optical fibers allows to achieve acquisition of opaqueness<sup>187</sup>.

As an alternative, the crushed or powdered waste PMMA can be mixed with other polymers to produce composites with improved characteristics. For instance, higher impact strength and elongation at break were attained by mixing PMMA and waste PVC collected from waste electrical and electronic equipment<sup>188, 189</sup>, improved toughness by blending with polycarbonate and acrylonitrile butadiene styrene (ABS). Waste PMMA was also used as an additive for asphalt<sup>190, 191</sup>. Besides aforementioned approaches, PMMA can be processed by dissolution/precipitation methods related to mechanical recycling<sup>192</sup>.

### **7.2. Chemical re-/upcycling of PMMA**

The high production cost and the wide application of PMMA materials encourage the development of a more efficient recycling method. The chemical recycling approaches of PMMA waste involve gasification<sup>193, 194</sup>, depolymerization, including pyrolysis (thermal cracking)<sup>195</sup>, and thermal carbonization<sup>196, 197</sup>.

PMMA is a relatively high-cost polymer and recovery of the initial monomer is desirable and expedient<sup>198, 199</sup>. The most common depolymerization strategies are thermal or thermochemical processes<sup>200-202</sup>. Thermal depolymerization is conducted under heating in an inert atmosphere through radical mechanisms<sup>203-206</sup>. This method allows to produce methyl methacrylate monomer in large amounts<sup>163, 201</sup>. Through the various reactors, the most prominent is fluidized bed depolymerization at 450 °C due to the high yield of methyl methacrylate and absence of metal impurities and other byproducts<sup>207-210</sup>. The liquid product from thermal depolymerization of PMMA in a fluidized bed reactor consists of around 95-98 % of methacrylic acid, which can be purified and repolymerized<sup>211</sup>.

Despite the fact that the thermal depolymerization of PMMA is a technology that potentially permits recycling in a closed cycle, this process is still facing many challenges. One of the main problems is connected with contaminations and impurities arising from the collection of PMMA waste or formed during the reaction<sup>184, 211, 212</sup>. Moreover, thermal processes are highly energy consuming and generate a sufficient carbon footprint<sup>186</sup>. Such challenge can be partially overcome by depolymerization in supercritical methanol<sup>213</sup> or electrolysis of PMMA to produce H<sub>2</sub><sup>214</sup>.

## **8. Rubber**

### **8.1. Mechanical recycling of rubber**

Methods for disposing of old tires are depending on the final application of feedstock. The common technological processes include the crushing of tires after removal of fillers. Then, the obtained powder or granules are widely used in various fields, especially in construction and building. The particular applications of recycled rubber are reviewed recently, where the utilisation of styrene-butadiene rubber (SBR) in civil engineering, building materials, coatings, and fillers are highlighted<sup>215, 216</sup>. In general, the use of waste tires as a building material has great potential: the mechanical treatment allows to isolate not only rubber, but also merit steel or polymeric fibers<sup>216</sup>. The combination of crumb rubber with fibers is able to sufficiently reinforce the traditional concrete.

Steel and fibers can be used as additives in concrete<sup>217</sup> and crumb rubber can be used as fine or coarse aggregate<sup>216</sup>, while powdered rubber can be applied as a binder or filler in

concrete<sup>218, 219</sup>. A special attention is paid to polymer blends with PE, PP, PS and PVC due to the enhanced toughness of prepared composites<sup>220</sup>.

Another promising way to process waste rubber is through reactive extrusion<sup>221</sup>. This method is characterized by good performance, almost complete absence of solvent and relatively short processing time and inexpensiveness. However, the technology is limited by the expensive equipment and emission of volatile decomposition products.

Despite the advantages of mechanical recycling (for example, in terms of environmental safety and sustainability due to the higher consumption of recycled polymers), low compatibility and poor interfacial adhesion between rubbers and thermoplastics leads to poor mechanical properties of the resulting composite materials (especially elongation at break and toughness)<sup>222</sup>. Moreover, mechanical recycling leads to significant drop in the cost of material, which contrasts with modern principles of circular economy.

## 8.2. Chemical re-/upcycling of rubber

Through different strategies of rubber recycling, chemical processing, in particular pyrolysis, is considered as one of the most promising techniques<sup>223</sup>. Pyrolysis involves the thermal decomposition of a used tire at a high temperature to transform rubber into value-added products<sup>223</sup>. As a rule, the optimum temperature for the process is 400-450 °C. Also, catalysts such as zeolites, Al-MCM-41, Al-SBA-15, Al<sub>2</sub>O<sub>3</sub>, SiO<sub>2</sub>, Ca(OH)<sub>2</sub>, AlCl<sub>3</sub> are used<sup>223</sup>. The pyrolysis and subsequent activation of waste rubber yield carbon-based materials with a large surface area and pore volume<sup>215</sup>. Pyrolyzed rubber represents a base for carbon nanotubes (CNTs)<sup>223</sup> and other carbon-based materials for supercapacitors and batteries<sup>224-226</sup>, catalysts<sup>227-229</sup>, and adsorbents<sup>215, 223, 230</sup>. Moreover, pyrolyzed SBR – char – could serve as a catalyst for heterogeneous oxidation of alkenes<sup>227</sup> or oxygen reduction reaction<sup>228, 229</sup>.

Very recently, a new approach for the disposal of rubber was proposed<sup>231</sup>. Direct current arc discharge plasma allows to convert waste rubber to ultrafine carbon powder, avoiding a vacuum and gas equipment that simplify the design of a plasma reactor and reduce specific energy consumption.

However, the main limitation of the process is the poor reproducibility: the properties and composition of the resulting carbon material depend on many factors, such as method of pyrolysis, composition of pristine rubber, nature of additives, *etc.* In this regard, the study of the relationship between these factors and the characteristics of activated carbon would be particularly useful. Moreover, pyrolysis requires elevated temperatures and, consequently, high consumption of

energy with evident carbon footprint. Due to this reason, the recent findings in this field are concentrated on the functional upcycling of rubbers.

## **9. Polycarbonate (PC)**

### **9.1. Mechanical recycling of PC**

Mechanical recycling of PC waste involves extrusion or grinding and physical mixing of the processed PC with other materials to prepare composites with improved characteristics<sup>232</sup>. This strategy has mostly been applied to improve the physical and mechanical properties of different construction materials, such as gypsum and cementitious mortars by adding processed PC compact discs (CDs) or digital versatile disc (DVDs) waste<sup>233-235</sup>, and PC industrial waste<sup>236</sup> or blended with PET<sup>237</sup>.

Despite the promising physicochemical properties of PC, such as optical clarity, excellent thermal and flame resistance, high impact strength, and high stability<sup>238</sup>, mechanical recycling of PC waste can be difficult due to its softness, high melt viscosity, and notch sensitivity<sup>238</sup>. Another possible limitation is the leaching of toxic bisphenol A (BPA) and its short lifetime<sup>239</sup>. To overcome these limitations, PC waste can be used as a polymer matrix with waste silk fibers and poplar wood flour<sup>240</sup> or blended with maleic anhydride grafted ABS<sup>241</sup> to make hybrid composite materials.

Nevertheless, the presence of various additives or degradation of PC due to multiple reasons (*e.g.*, light, radiation, chemicals, *etc.*) leads to the deterioration in properties of the recycled materials and hinders mechanical recycling<sup>242</sup>.

### **9.2. Chemical re-/upcycling of PC**

Chemical recycling of PC can be performed in the presence of some impurities, which do not deteriorate the quality of the final product but sufficiently affect the reaction rate<sup>243,244</sup>. Mostly BPA polycarbonate from panels, windows, and even disks can undergo chemical re-/upcycling<sup>245</sup>. Similar to PET, hydrolysis<sup>246,247</sup>, alcoholysis<sup>248</sup>, aminolysis<sup>249</sup>, pyrolysis<sup>250</sup>, hydroglycolysis<sup>251</sup> and hydrogenolysis<sup>252</sup> were applied to get pure BPA (recycling) or a variety of products (upcycling).

Pyrolysis requires harsh reaction conditions ( $> 300\text{ }^{\circ}\text{C}$ ) and proceeds unselectively with formation of different by-products (BPA, mixture of light-weight molecules, phenolic compounds and condensation products) including greenhouse gases and 20-30 wt.% low-value char<sup>253</sup>. The use of catalysts or additives, such as metal chlorides (Fe and Cu<sup>254</sup>, Sn and Zn<sup>255</sup>), basic oxides

(Ca, Mg)<sup>256</sup> decrease the reaction time and temperature, reduce amount of char and improve selectivity. However, yields of PC pyrolysis are commonly quite low and formed carbonaceous materials require specific separation and purification procedures<sup>254, 255</sup>. Therefore, additional time and energy consuming purification steps require for the isolation of value-added products.

Alternative option for PC re-/upcycling is the cleavage of O–CO bond *via* nucleophilic substitution. Such approach is especially effective in combination with nucleophilic solvents such as water or alcohols. Indeed, the reaction with water as the most environmentally friendly solvent, gives phenol and CO<sub>2</sub>. However, the hydrolysis requires the addition of concentrated acids or bases in large quantities, or, alternatively, changing the reaction conditions to supercritical for phase compatibility<sup>257</sup>. Various catalysts, such as acids<sup>258</sup> or metal oxides, hydroxides and triflates (alkali, alkaline earth, rare earth)<sup>259, 260</sup> are using to facilitate PC hydrolysis. Catalytic conditions are usually milder and give BPA with higher yields in comparison with stoichiometric. However, the application of acids or bases is connected with considerable expenses on the utilisation or recyclization of reagents together with specific requirements to technological apparatus and pipelines. Indeed, CO<sub>2</sub> evolving during process sufficiently affect the carbon footprint of technology.

In contrast to water, alcoholysis allows to avoid CO<sub>2</sub> release during PC cleavage by methanol, ethanol, phenol, diol<sup>243, 261</sup>. In this case, PC represents an exceptional alternative to toxic phosgene, carbon monoxide and their derivatives as carbonyl sources to maximize green metrics in PC upcycling. Despite the clear advantages of alcoholysis, the alcoholysis needs the addition of common organic bases 1,8-diazabicyclo(5.4.0)undec-7-ene (DBU), 1,5,7-triazabicyclo[4.4.0]dec-5-ene (TBD), which reusability is a still questionable.

In addition to alcoholysis, the nitrogen or sulphur-based nucleophiles can also be applied for the preparation of valuable urea, thiourea, thiocarbonate and carbamate derivatives or even new polymer (PUs) as an attractive alternative<sup>249, 262, 263</sup>. Indeed, the technological realization of such processes requires the careful optimization of conditions including solvent and reagent ratio. For instance, Nacci *et al.* developed bifunctional acid/base catalyst for PC upcycling composed of nanostructured zinc oxide and tetrabutylammonium chloride (ZnO-NPs/Bu<sub>4</sub>NCl) serving as Lewis acid and base, respectively<sup>249</sup>. The application of developed catalyst led to smooth depolymerization of PC in the presence of N-centered nucleophiles furnishing the corresponding ureas. Alternatively, these compounds can be prepared by reaction of phosgene with primary or secondary amines, by direct metal-catalysed oxidative carbonylation of amines<sup>264</sup>.

Another promising example of chemical upcycling was reported by Sardon *et al.*, where PC was treated by diols at 160 °C in the presence of TBD and methanesulfonic acid with formation

of BPA innovative carbonate-containing diols<sup>265</sup>. Carbonate-linked diols were subsequently polycondensated to get solid polymer electrolytes for batteries with considerable ion conductivity.

There are a few experimental strategies to make solvolysis reaction more environmentally friendly. The first approach is the addition of cosolvent, such as THF, 1,4-dioxane, 1,2-dichloroethane, toluene, *N*-methyl-2-pyrrolidone, which assist in swelling or dissolving polycarbonate. Alternatively, ionic liquids can be considered as desirable reaction media to promote swelling and dissolution of PC together with considerable catalytic activity promoting the solvolysis<sup>266</sup>. A further improvement is switching to microwave heating for energy saving<sup>267</sup>. Despite some improvement in this field, the use of hazardous solvents and related costs are limiting scaling up re-/upcycling process.

Finally, the PC chains were cleaved by simple hydrogenation in the presence of transition metals as a catalyst (Fe, Mn, Co, Ru, Ir)<sup>268,269</sup> with the formation of BPA and methanol as products, where reaction mixtures that can easily be recovered. In addition to expensive metals, temperature higher than 120 °C and pressure around 60-100 bar (H<sub>2</sub>) is required to get high yields and cannot be adapted to the industry level now.

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