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Room Temperature Organocatalyzed Reductive Depolymerization of Waste Polyethers, Polyesters, and Polycarbonates

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The reductive depolymerization of a variety of polymeric materials based on polyethers, polyesters, and polycarbonates is described using hydrosilanes as reductants and metal-free catalysts. This strategy enables the selective depolymerization of waste polymers as well as bio-based polyesters to functional chemicals such as alcohols and phenols at room temperature. Commercially available $B(C_6F_5)_3$ and $[Ph_3C^+,B(C_6F_5)_4^-]$ catalysts are active hydrosilylation catalysts in this procedure and they are compatible with the use of inexpensive and air-stable polymethylhydrosiloxane and tetramethyldisiloxane as reductants. A significant advantage of this recycling method is derived from its tolerance to the additives present in waste plastics and its ability to selectively depolymerize mixtures of polymers.

Thanks to their relatively low cost and versatile properties, polymeric materials have found numerous market applications in various fields, such as electronics, home appliances, cars, or medicine. In particular, the global production of plastics, including polyolefins, polyamides, polyesters, has increased from 1.7 to 280 million tons over the last 60 years.[1] This widespread utilization generates millions of tons of waste plastics each year. Because the cost for recycling waste polymers often exceeds the production of new materials from raw feedstocks, the sustainability of this industrial segment could be largely improved through a better end-of-life management and the development of efficient recycling methods, in agreement with changes in legislation/regulation.[2] In 2012, Europe generated 25.2 Mtons of plastic waste among which only 26 % were recycled, mostly by mechanical methods. Yet, in January 2014, the European Parliament has adopted a resolution to increase recycling rates and reduce incineration and landfill disposal of plastics to suppress discharge of plastics in Europe by 2020.[3]

In this context, the development of selective depolymerization methods that can be used to break down waste polymers to valuable monomers or chemicals appears a promising strategy. In particular, polyesters and polycarbonates, which are commonly utilized in construction, automotive, packaging, and agricultural industries, were the focus of recent efforts, and their chemical recycling has been proposed based on hydrolysis, transesterification and transcarbonation reactions.[4] Nonetheless, these methods still suffer from harsh reaction conditions and low selectivities, which stem from the low solubility of plastic materials together with the presence of additives or polymer mixtures in the waste material matrix. Alternatively, C–O bond cleavage induced by reduction methods could promote the depolymerization of oxygenated plastics, such as polyethers, polyesters, and polycarbonates, and at the same time yield energetic and valuable alkanes and alcohols as products. Yet, this strategy remains largely underdeveloped, and only recently Robertson et al. have demonstrated the first examples of reductive depolymerization of polyesters and polycarbonates with Ru catalysts.[5] Although this method has the advantage of using H_2 as a cost-efficient reductant, disadvantages are elevated reaction temperature ($>120\,^{\circ}C$) and H_2 pressure (>50 bar) and the use of noble-metal catalysts. Reasoning that the mild reduction potential of the Si–H linkage in hydrosilanes combined with the strong affinity of Si atoms to O atoms could facilitate the depolymerization of oxygenated materials,[6] we describe herein the unprecedented depolymerization of polyethers, polyesters, and polycarbonates under hydrosilylation conditions. This method has the advantage of using $B(C_6F_5)_3$ as a metal-free catalyst and it operates successfully at room temperature for the recycling of mixtures of polymers as well as waste materials derived from post-consumer products.

$B(C_6F_5)_3$ is a well-established hydrosilylation catalyst that has been successfully utilized in the reduction of carbonyl derivatives, alcohols, and ethers.[7] Moreover, this metal-free Lewis acid enables the utilization of tetramethyldisiloxane ($Me_2SiHOSiHMe_2$, TMDS), and polymethylhydrosiloxane [$Me_3Si(OSiMeH)_nOSiMe_3$, PMHS] reductants, which are air stable, non-toxic, and cost-efficient byproducts of the silicone industry (2–5 € per mol).[8] The reductive deoxygenation of polyethylene glycol (PEG) was thus achieved by using 2.3 equiv PMHS and 2 mol % $B(C_6F_5)_3$ as a model system for the depolymerization of polyethers (Table 1). Within 1 h at 25 °C in CH_2Cl_2, 94 % of the PEG starting material is deoxygenated, leading to the formation of ethane (and siloxane byproducts), as observed by ^1H NMR and GC analyses. Ethane being the only organic product observed by decomposition of PEG, its yield was approximated by the conversion of the starting materials. Similarly, using 1.1 equiv TMDS per ether linkage, PEG was completely converted to ethane. Notably, no intermediate could be observed in the reduction of PEG to ethane using PMHS and TMDS, including a default hydrosilane (see the Sup-

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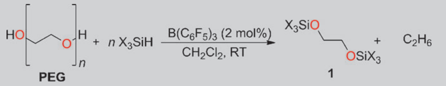
Table 1. PEG depolymerization promoted by $B(C_6F_5)_3$. Product **1** yields were obtained by GC–MS analysis after calibration. Ethane yields were deduced from the total conversions provided by ^1H NMR analysis.

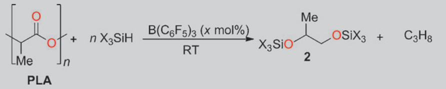


Silane	Equivalents	Reaction time [h]	Yield [%] 1	Yield [%] C_2H_6
PMHS	2.3	1	0	94
TMDS	1.1	1	0	>99
Et_3SiH	1.3	3	41	58
	2.3	16	0	90

porting Information). The depolymerization of PEG was thus carried out using Et_3SiH as a reductant to preserve functional groups and access building blocks with greater economic value. Previous reports on the reduction of carboxylic acids and ether derivatives have indeed shown that Et_3SiH is a milder reductant than PMHS and TMDS in the presence of $B(C_6F_5)_3$.[9] The reaction of PEG with only 1.3 equiv Et_3SiH per ether unit results in the complete depolymerization of the polyether within 3 h and selectively affords **1** in 41 % yield and ethane as a byproduct. Interestingly, **1** is the silylated form of ethylene glycol and its formation suggests that the reduction of two consecutive C–O bonds in the polyether proceeds non-symmetrically. Although silylated ethanol, $Et_3SiOC_2H_5$, would be expected for the regular transfer of the Si–H hydride to the carbon atom at the β-position of a Et_3SiO siloxy group, the depolymerization of PEG occurs through the selective transfer of two hydrides to vicinal carbon atoms to yield ethane and **1** in a 1:1 molar ratio. This finding was further confirmed by the formation of **1** using labelled Et_3SiD (see the Supporting Information). This selectivity likely derives from different steric repulsions between the polyether chain and an ethyl group.

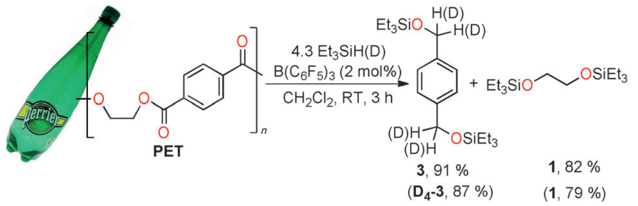
The selective reductive depolymerization of more oxidized polymer matrices is a more challenging issue, and the hydrosilylation of polyesters was thus explored. Using 5 mol % $B(C_6F_5)_3$, a commercial sample of racemic polylactic acid (PLA) was reacted with 3.3 equiv Et_3SiH in CH_2Cl_2. Within 16 h at 25 °C, the catalytic depolymerization of PLA afforded **2**, as a racemic mixture, in 65 % yield (Table 2). This observation is consistent with previous reports showing that the $B(C_6F_5)_3$-catalyzed hydrosilylation of esters affords silylethers.[7e] Interestingly, the use of 2.0 equiv TMDS led to the full conversion of PLA (>99 %) to propane after 1 h in CH_2Cl_2, whereas benzene must be utilized as a solvent with PMHS to avoid gel formation (Table 2).

Table 2. PLA depolymerization promoted by $B(C_6F_5)_3$. Product **2** yields were obtained by GC–MS analysis after calibration. Propane yields were deduced from the total conversions provided by ^1H NMR analysis.



Silane	Equivalents	Solvent	Reaction time [h]	Catalyst loading [mol %]	Yield [%] 2	Yield [%] C_3H_8
Et_3SiH	3.3	CH_2Cl_2	16	5	65	0
TMDS	2.0	CH_2Cl_2	1	2	0	>99
PMHS	4.0	C_6H_6	1	2	0	56
Et_3SiH	8.0	CH_2Cl_2	24	10	36	n.d.

Unlike PLA, polyethylene terephthalate (PET) is not biodegradable and is also less reactive in hydrolysis or transesterification reactions.[10] PET is a widespread thermoplastic polymer mostly utilized in textile applications (polyester) and bottle packaging.[11] The depolymerization of PET was thus undertaken using waste Perrier bottles collected from a recycle bin. Despite the presence of additives such as a green dye, PET is efficiently depolymerized at room temperature using 4.3 equiv Et_3SiH and 2 mol % $B(C_6F_5)_3$ to afford two disilylethers, namely **1** and **3** (Scheme 1). The reaction is carried out at room temperature and did not require any other additive. **1** and **3** are readily purified by column chromatography and isolated in 72 % and 85 % yield, respectively. Importantly, ethylene glycol and 1,4-phenylenedimethanol can be obtained quantitatively by hydrolysis of **1** and **3**, respectively, using 2.1 equiv TBAF·3 H_2O (see the Supporting Information). Notably, 1,4-phenylenedimethanol is a valuable building block that can be oxidized back to terephthalic acid or can undergo Friedel–Crafts alkylation to directly yield hypercross-linked polymers (HCPs).[12] 1,4-Phenylenedimethanol can also serve as an intermediate for the production of other useful chemicals, namely terephthalaldehyde, phthalonitriles, and 1,4-bis(bromomethyl)-benzene, currently used as intermediates in the production of plastics, pesticides, perfumes, dyes, and fluorescent brighteners.[13]

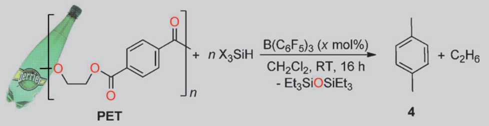


Scheme 1. PET depolymerization using Et_3SiH.

The use of 11.0 equiv PMHS and 6.0 equiv TMDS as reductants in the depolymerization of PET results in the production of *p*-xylene (**4**) in >75 % yields and ethane. When PMHS is used, a larger quantity of solvent is needed to prevent gel formation (see Table 3 and the Supporting Information).

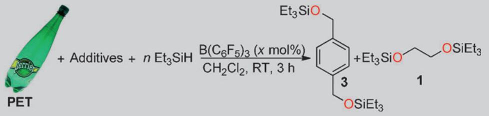
The depolymerization of PET using Et_3SiH, similarly to that of PLA, produced disilylethers. This suggests that in both cases the two hydride transfers from silicon proceeded on the carbon bearing the carbonyl function, in agreement with the formation of **D_2-2** and **D_4-3** from Et_3SiD (see the Supporting Information Scheme 1). Nevertheless, the depolymerization reaction of PET is faster and requires a lower catalyst loading (2 mol %) than that of PLA (5 mol %). This effect may be due to the increased positive charge on the carbon atom bearing the ester function in PET. This difference in reactivity can provide a selective cleavage of PET in the presence of PLA. In fact,

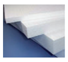
Table 3. PET depolymerization using PMHS and TMDS and $B(C_6F_5)_3$ as catalyst. Product **4** yields were obtained by GC–MS analysis. Ethane yields could not be determined by ^1H NMR analysis owing to the insolubility of PET in CH_2Cl_2.



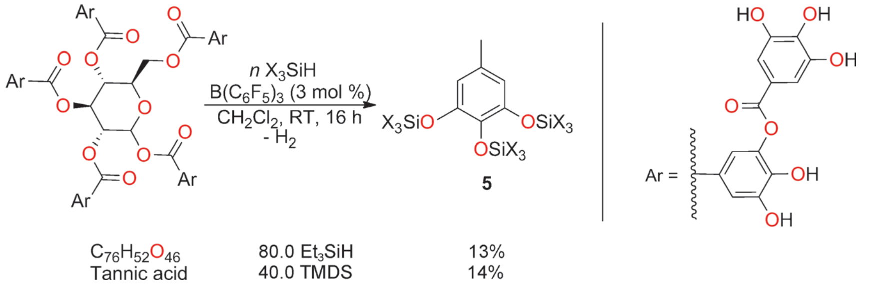
Silane	Equivalents	Catalyst loading [mol %]	Yield of **4** [%]
TMDS	6.0	5	82
PMHS	11.0	7.5	75
Et_3SiH	6.3	7.5	49

Table 4. PET depolymerization promoted by $B(C_6F_5)_3$ in the presence of other polymeric materials.



	Additive	Equivalents	PET equiv.	Catalyst loading [mol %]	Yield [%] **3**	Yield [%] **1**
	PLA	1	4.2	2	51	62
	PS	1	5.2	5	66	69
	PS + PVC	1 + 1	5.2	5	65	61

when using 4.2 equiv Et_3SiH and 2 mol % $B(C_6F_5)_3$, PET is selectively depolymerized, from a mixture of PLA and PET, to **1** and **3**, leaving PLA unmodified (Table 4). Changing the operating conditions to 7.2 equiv Et_3SiH and 10 mol % $B(C_6F_5)_3$, the depolymerization of both polymers can be achieved in 16 h (see the Supporting Information). On the other hand, waste polymeric materials may contain other types of polymeric materials such as polystyrene (PS) and/or polyvinyl chloride (PVC).[14] Consequently, the hydrosilylation reaction was conducted using equimolar mixtures of PET + PS (expanded) and PET + PS + PVC (extracted from commercial PVC pipelines). In both cases, the presence of other polymeric materials had no influence on the outcome of the reaction and only PET depolymerization products were observed after 3 h, with > 61 % yields (Table 4). Although the detailed practical aspects of this depolymerization strategy are still to be explored, these results already tackle a strong limitation usually encountered with the recycling of complex mixtures of waste polymers.

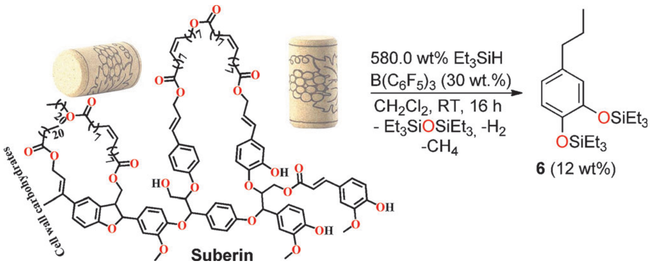


Scheme 2. Tannic acid depolymerization using Et_3SiH and TMDS.

Having in hand a depolymerization method compatible with the presence of impurities, we then explored its potential in the depolymerization of biobased polyesters such as suberin and hydrolysable tannins. Indeed, the reduction of the ester linkages in these biomass products could afford an entry to renewable aromatic molecules with high added value. In particular, reduction of tannic acid with 80.0 equiv Et_3SiH and 3 mol % $B(C_6F_5)_3$ enables the formation of a single aromatic compound, **5**, in 13 % yield after 16 h at RT (Scheme 2). The depolymerization of tannic acid can also be carried out using 40.0 equiv TMDS as a hydride donor to obtain **5** in 14 % yield, based on the initial quantity of aromatic units in tannic acid.

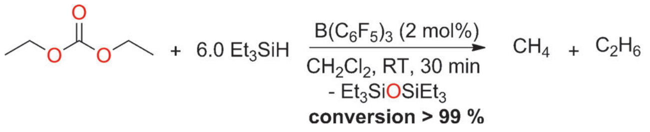
Being the main component of cork, suberin is another example of a natural polyester. Cork-based French wine bottle stoppers were finely ground and dried overnight in vacuo as a source of suberin. Exposing this material to a CH_2Cl_2 solution containing 580.0 wt % Et_3SiH and 30 wt % $B(C_6F_5)_3$ resulted in a substantial solubilization of the insoluble cork matrix, and GC–MS analysis of the reaction mixture after 16 h at RT showed the presence of a complex mixture of products. Nevertheless, the silylated catechol derivative **6** was clearly identified (Scheme 3). Although **6** was not isolated, its yield was determined by GC–MS analysis after calibration with an authentic sample, showing that 12 wt % suberin was successfully converted to **6** by reductive depolymerization.

The $B(C_6F_5)_3$-catalyzed hydrosilylation reaction being a potent methodology for the depolymerization of synthetic and natural polyesters, its potential was assessed in the reduction of polycarbonates. To the best of our knowledge, the reduction of organic carbonates using $B(C_6F_5)_3$ and hydrosilanes has not been reported so far although metal catalysts have



Scheme 3. Suberin depolymerization using Et_3SiH. Hydrosilane and catalyst quantities were calculated based on the initial mass of suberin. Suberin structure was obtained by Kolattukudy.[15]

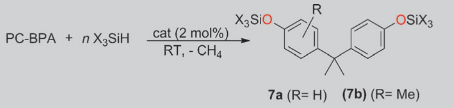
been recently used in the reductive amination of carbonates.[16] Moreover, base-catalyzed metal-free depolymerization of polycarbonates was lately described by Darensbourg et al.[17] Addition of 6.0 equiv Et_3SiH and 2 mol% $B(C_6F_5)_3$ to a CH_2Cl_2 solution of diethyl carbonate resulted in a rapid gas evolution, and 99% of the starting materials were converted to methane, ethane, and siloxanes within 1 h at RT (Scheme 4).



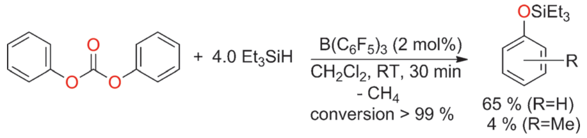
Scheme 4. Reductive hydrosilylation of diethylcarbonate using Et_3SiH as reducing agent.

This result prompted us to test the reductive depolymerization of bisphenol-A polycarbonate (PC-BPA). Although PC-BPA is being banned from food-contact applications, it remains widely utilized in data storage (CDs, DVDs) and armored windows. Hydrosilylation of PC-BPA using 2 mol% $B(C_6F_5)_3$ in the presence of 4.2 equiv Et_3SiH led to the formation of disilylated BPA (**7 a**) in 82% yield and methane (Table 5). Interestingly, a side product (**7 b**, 28% yield), which formally results from the methylation of **7 a**, was detected by GC–MS analysis of the crude mixture. The extra methyl group in **7 b** likely derives from the reduction of the carbon atom initially bearing the carbonyl function in PC-BPA. It has indeed been observed that aromatic derivatives can undergo Friedel–Crafts alkylation under electrophilic hydrosilylation conditions.[9a, 18] To test this hypothesis and rule out the possible involvement of impurities in the PC-BPA starting material in the formation of **7 b**, the reduction of diphenyl carbonate was carried out, using Et_3SiH and $B(C_6F_5)_3$ as catalyst. As depicted in Scheme 5, even though triethyl(phenoxy)silane was the main product in the hydrosilylation of diphenyl carbonate (65%), a methylated side product was also obtained in 4% yield.

Table 5. PC-BPA depolymerization using Et_3SiH, TMDS, and PMHS.



Silane	Equivalents	Solvent	Reaction time [h]	Catalyst	Yield [%] 7a	Yield [%] 7b
Et_3SiH	4.2	CH_2Cl_2	1	$B(C_6F_5)_3$	82	28
TMDS	2.2	CH_2Cl_2	1	$B(C_6F_5)_3$	98	0
PMHS	4.2	CH_2Cl_2	1	$B(C_6F_5)_3$	gel formation	
Et_3SiH	4.2	C_6H_6	16	$[Ph_3C^+,B(C_6F_5)_4^-]$	47	26



Scheme 5. Reductive hydrosilylation of diphenylcarbonate using Et_3SiH as reducing agent.

The hydrosilylation of PC-BPA appears to be more rapid and less selective than the reduction of polyethers and polyesters. With only 1.0 equiv Et_3SiH, methane evolution was still observed and no intermediate could be characterized. As expected, TMDS is a powerful reductant in the depolymerization of BPA-PC and **7 a** is obtained in 98% yield, using 2.2 equiv TMDS after 1 h at RT (Table 5). In contrast, the formation of a gel is noticed in the presence of PMHS despite the use of benzene. Although it is known that $[Ph_3C^+,B(C_6F_5)_4^-]$ is inefficient in the hydrosilylation of alcohols, ethers, and esters, it is notable that this trityl salt can replace the $B(C_6F_5)_3$ organocatalyst in the reduction of carbonates. Indeed, **7 a** is formed in 47% yield after 16 h through depolymerization of PC-BPA with 4.2 equiv Et_3SiH and 2 mol% $[Ph_3C^+,B(C_6F_5)_4^-]$. $[Ph_3C^+,B(C_6F_5)_4^-]$ is thus somewhat less reactive than $B(C_6F_5)_3$ and it is also less selective as **7 b** represents 36% of the final product mixture [vs. 28% using $B(C_6F_5)_3$; Table 5].

To summarize, we have disclosed herein the first depolymerization of a wide range of polymeric materials based on polyethers, polyesters, and polycarbonates under metal-free conditions. Using $B(C_6F_5)_3$ or $[Ph_3C^+,B(C_6F_5)_4^-]$ as organocatalysts, a variety of functional chemicals can be selectively obtained by tuning the reaction conditions and the nature of the hydrosilane reductant. Notably, the inexpensive and air-stable hydrosilanes polymethylhydrosiloxane (PMHS) and tetramethyldisiloxane (TMDS) are able to reduce polyethers, polyesters, and polycarbonates, including waste materials, as well as biobased polyesters, at room temperature. A significant advantage of this recycling method derives from its tolerance to the additives present in waste plastics and its ability to selectively depolymerize mixtures of polymers.

Experimental Section

Supplementary schemes, detailed descriptions of experimental methods, and results are provided in the Supporting Information.

Acknowledgements

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Keywords: depolymerization · homogeneous catalysis · hydrosilylation · polyesters · recycling

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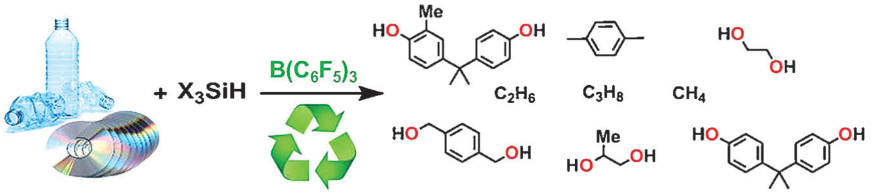
Room Temperature Organocatalyzed Reductive Depolymerization of Waste Polyethers, Polyesters, and Polycarbonates



Silanes pitted against waste: The reductive depolymerization of a variety of polymeric materials based on polyethers, polyesters, and polycarbonates is described, using hydrosilanes as reductants and metal-free catalysts. This strategy enables the selective depolymerization of waste polymers as well as bio-based polyesters to functional chemicals such as alcohols and phenols at room temperature.