

## ARTICLE

# Investigation of the decomplexation of polyamide/CaCl<sub>2</sub> complex toward a green, nondestructive recovery of polyamide from textile waste

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**Abstract**

The ‘circular economy’ concept envisages deriving the maximum value out of resources and reducing waste to a minimum. In textiles, that includes the recovery of fiber materials out of used clothing and reusing them in the construction of new clothes. Processes such as mechanical separation, depolymerization treatment, and selective dissolutions of individual polymers are applied. We investigate the approach of selective nondestructive dissolution and recovery of polyamide fiber from mixed textile waste by using the solvent system CaCl<sub>2</sub>/ethanol/water (CEW) based on complexation and decomplexation of polyamide (PA). The results show that PA is precipitated and decomplexed by simple addition of water and a substantial amount of previously incorporated calcium by complexation, is removed. The recovered polyamide shows properties similar to pristine polyamide. Investigation on a mixed textile waste model of polyamide/wool demonstrates that CEW treatment can successfully separate different fiber materials. The nondestructive approach in dissolving PA using CEW, clearly shows the benefit, that PA fiber can be separated by controlled complexation/decomplexation without degradation, thus avoiding the repolymerization step. Furthermore, the solvent system is made of abundantly available materials that are inexpensive and used widely in industrial-scale operations. Thus, the concept will make significant contribution to a green textile recycling approach.

**KEYWORDS**

polyamides, recycling, separation techniques, textiles

## 1 | INTRODUCTION

The landscape of clothing manufacture and use has changed dramatically in the past two decades. Globally, the

production of clothing has doubled but average usage has reduced by about a third.<sup>1</sup> Some of the discarded clothing is resold as second-hand goods, but the major proportion is either incinerated or disposed in landfills, and there is

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very little recycling of the materials. In Europe, around 4.3 million tons of textile waste is disposed annually due to the lack of viable recycling strategies.<sup>2</sup> Furthermore, fiber production consumes significant amounts of raw materials, energy, and water, and therefore the current system is wasteful of resources and detrimental to the environment.

One of the concepts for better utilization of raw materials is the recycling of the polymers from waste clothing back into the manufacture of new textiles. Very often, clothing textiles are, however, not single-component products but mixtures or blends of different polymer types, for example, polyester with cotton, or wool with polyamide. Effective recycling requires a complete separation and recovery of all polymers from blends, without damage to any. That is difficult to achieve with mechanical processes as the components in blends are mixed intimately. Thus, the chemical dissolution of individual polymers is an attractive option.<sup>3</sup>

As cellulose, polyester, and polyamide represent the majority of polymer types found in textiles, many research efforts are put into the chemical separation and recovery of these polymers. Recent work on chemical recycling of cellulose from textile wastes is based on the utilization of amine oxide solvents, for example, *N*-methyl morpholine *N*-oxide (NMMO)<sup>4,5</sup> or ionic liquids (IL).<sup>6–8</sup> For example, 1,5-diazabicyclo[4.3.0]non-5-ene acetate is investigated as direct solvent to selectively dissolve the cotton part in cotton polyester blended textile waste.<sup>7</sup> The cellulose solution is then spun to new man-made fibers, including the remaining dyestuff from the textile wastes.<sup>8</sup> The strategy for chemical recycling of polyester focuses on the depolymerization of polyester, yielding its precursor monomers, for example, terephthalic acid and ethylene glycol,<sup>3</sup> for example by thermal and catalytic decomposition,<sup>9,10</sup> hydrolysis<sup>11</sup> or methanolysis.<sup>12</sup> Different strategies have also been proposed for polyamides including hydrolysis,<sup>13–15</sup> supercritical fluids,<sup>16–18</sup> ionic liquids under microwave irradiation,<sup>19</sup> and ring-closing reactions to produce *N*-acetylcaprolactam.<sup>20</sup>

While the depolymerization strategy works best for single polymer synthetic products, it requires, however, extensive purification steps to separate and isolate the different monomers before repolymerization in the case of fiber mixtures as very often observed in textiles. Thus, different strategies need to be developed for the separation and recovery of the fiber components.

In this work, we follow the approach of nondestructive dissolution and recovery of polyamide fibers and report on investigations to dissolve out and recover polyamide (PA) by the controlled complexation/decomplexation without degradation. In general,

polyamides dissolve in solvents such as phenol, *m*-cresol, and formic acid, but they are unsuitable for textile recycling purposes. One problem is of handling large volumes of organic solvents in commercial-scale operations, and another is solubility and stability of other components such as wool in solvents such as formic acid. Hence, there is a pressing need in finding alternative, environmentally friendly solvent systems for recycling of PA containing mixed textile waste. The solvent employed in our investigations is a mixture of CaCl<sub>2</sub>, ethanol, and water (CEW). We have previously investigated the dissolution and swelling action by complexation.<sup>21,22</sup> However, another very important requirement for a possible application of this solvent system for the recovery of PA from textile waste is the intake polymer structure of the dissolved and reprecipitated polymer. This can only be achieved through a proper decomplexation process leading to PA free of Ca<sup>2+</sup> in the ideal case. Thus, the focus of the present work is the investigation of the decomplexation process as one of the key steps in the recovery and recycling of polyamide from mixed textile waste with the specific focus to remove solvent residues, that is, calcium, from the recovered PA.

## 2 | EXPERIMENTAL

### 2.1 | Materials

The fiber materials used in the work were pre-cleaned polyamide 66 (PA66) staple fibers of average diameter  $15.7 \pm 0.4 \mu\text{m}$  and ca. 3 cm in length, and merino wool fibers of average diameter 15–30  $\mu\text{m}$  and 15 cm in length, kindly provided by Schoeller GmbH & Co. KG, Austria. It is worth noticing that as a natural fiber, merino wool does not exhibit a uniform diameter along its length. Calcium chloride dihydrate ( $\geq 99\%$ ), ethanol (99.9%vol), and deionized water (conductivity  $< 10 \mu\text{S cm}^{-1}$ ) were used in formulating the CEW mixture (i.e., polyamide solvent) to contain CEW in mole fractions (%) of 12.50, 62.50, and 25.00 respectively. All other reagents were of analytical grade. An ordinary kitchen sieve, with  $19 \times 15$  holes per  $\text{cm}^2$  of average dimensions  $0.667 \times 0.419 \text{ mm}^2$  each, was used to filter the precipitated polymer particles.

### 2.2 | Dissolution procedure and sample preparation

In this study, we used a model mixture consisting of 50 wt% PA66 and 50 wt% wool fibers to simulate a textile product with mixed fibers. The fiber mixture model is chosen in order to exclude the influence of yarn and

textile construction on the dissolution process as our focus was the decomplexation of the dissolved polyamide part. For the mixture model, PA and wool fibers were mixed thoroughly by hand. The fiber mixes (5 g each) were immersed in separate 85 g portions of the CEW mixture and agitated on a shaking platform at room temperature for 60 min to completely dissolve the polyamide. Then the slurry was filtered through a sieve to separate wool from the dissolved polyamide. The wool residue on the sieve was rinsed with deionized water to wash off solution and solvent residues, dried at room temperature, and weighed.

For the decomplexation study, 200 ml deionized water (nonsolvent) was added to the filtrate containing the dissolved PA, whereupon the polymer reprecipitated and slurries were formed, the solutions were agitated on a shaking platform to rinse the reprecipitated polymer. There were six rinsing periods: 0, 5, 10, 20, 30, and 45 min. At the end of each period, the reprecipitated polyamide was recovered by filtering the slurries through a sieve, and the samples were labeled sequentially S1 through to S6. For 0 min of rinsing (i.e., sample S1), the slurry was stirred once with a glass rod after addition of the 200 ml deionized water, and filtered through the sieve immediately. All filtered reprecipitates when on the sieve, were washed once with 50 ml deionized water, placed to dry in a laboratory oven at 105 °C for 4 h, and then rested in a desiccator over powdered P<sub>2</sub>O<sub>5</sub> before further analyses.

### 2.3 | Analyses of separated wool fiber and reprecipitated polymer

The difference between initial mass of fiber mixes and the final mass after polyamide dissolution was used as a measure of polyamide mass loss. The residual wool fibers were examined for evidence of damage under a light transmission optical microscope (CX 41, Olympus Corporation, Japan).

Attenuated total reflectance Fourier transform infrared spectra (FTIR-ATR) of the original and reprecipitated polyamide were recorded on a Bruker Vector 22 FTIR spectrometer (Bruker Corporation, USA) equipped with a MIRacle™ Horizontal ATR accessory fitted with a diamond crystal double reflection universal plate, from PIKE Technologies (USA). A total of 32 scans were collected per measurement, from 500 to 4000 cm<sup>-1</sup> at a resolution of 4 cm<sup>-1</sup>.

Thermal analysis of the original and reprecipitated polyamide was performed on a differential scanning calorimeter (DSC3, Mettler Toledo, USA) on about 8 mg specimen encased in 40 µl aluminum crucibles with a pierced

lid, under 50 ml min<sup>-1</sup> nitrogen flow in the temperature range of 20–280°C, at heating and cooling rate of 10°C min<sup>-1</sup>. A total of four measurements were performed per sample, and the results were analyzed with the on-board evaluation software (Mettler STAre, Version 15.01).

The calcium content on the original and reprecipitated samples was quantified through complexometric titration with 0.01 mol L<sup>-1</sup> EDTA. About 0.1 g of specimen were extracted in 100 ml of 0.1 M HCl at 60°C for 30 min. A volume of 50 ml extract (the analyte) was mixed with 5 ml of NH<sub>4</sub>Cl/NH<sub>3</sub> buffer at pH 10–11. The buffer was comprised of 1.26 mol L<sup>-1</sup> NH<sub>4</sub>Cl, 7.64 mol L<sup>-1</sup> NH<sub>3</sub> as well as 0.5 g L<sup>-1</sup> EDTA-magnesium-dipotassium salt (Titriplex®). A solution of the metallochromic indicator Eriochrome Black T was made up by dissolving 0.2 g of the indicator in 15 ml of 25% NH<sub>3</sub> and 5 ml anhydrous ethanol, and five drops of this solution were added to each analyte. The titrations were performed on an auto-titrator (DL50, Mettler Toledo, USA) equipped with a phototrode (DP550, Mettler Toledo, USA) at incremental steps of 0.02 ml. Two replicate titrations were performed per sample, and the calcium content was calculated from Equations (1) and (2):

$$c_{\text{Ca}} = \frac{V_{\text{EDTA}} \cdot c_{\text{EDTA}}}{V_{\text{Analyte}}}, \quad (1)$$

$$\frac{m_{\text{Ca}}}{m_{\text{Specimen}}} = \frac{c_{\text{Ca}} \cdot V_{\text{Extract}} \cdot M_{\text{Ca}}}{m_{\text{Specimen}}}, \quad (2)$$

where,

$c_{\text{Ca}}$ ,  $c_{\text{EDTA}}$  = concentrations of calcium and EDTA in extract and titrant respectively (mol L<sup>-1</sup>).

$V_{\text{EDTA}}$ ,  $V_{\text{Analyte}}$ ,  $V_{\text{Extract}}$  = volumes of EDTA (titrant), analyte, and extract (L).

$M_{\text{Ca}}$  = molar mass of calcium (g mol<sup>-1</sup>).

$m_{\text{Ca}}$ ,  $m_{\text{Specimen}}$  = mass of calcium and polyamide specimen respectively (g).

## 3 | RESULTS AND DISCUSSION

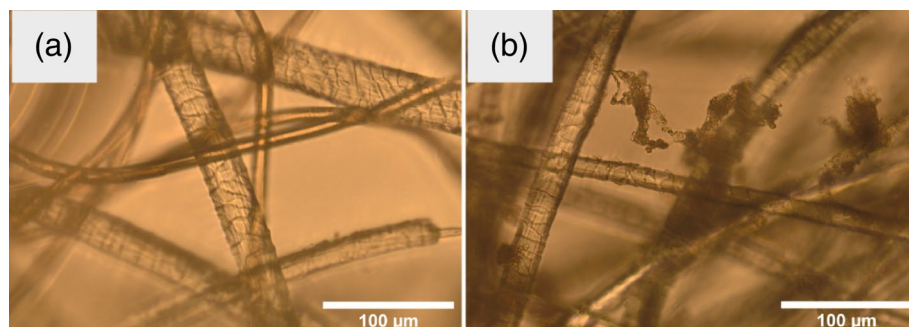
### 3.1 | Analysis of separated wool fibers

It had previously been determined that CEW mixtures do not dissolve wool or measurably reduce its mass without presence of a cysteine reducing agent.<sup>23</sup> That was confirmed in this work as well, and it may be observed in Figure S1 that no differences can be found between untreated wool and the fiber after treatment.

The initial mass of the fiber mixture and their final mass after CEW treatment, water rinsing, and drying are shown in Table 1. The change in mass was attributed

**TABLE 1** The proportion of polyamide and wool in fiber mixes, their initial and final masses, and the mass loss expressed as percent of initial polyamide amount in the mixes

Proportion of polyamide/wool in fiber mix (mass %)	Initial mass of fiber mix (g)	Final mass of fiber mix after polyamide dissolution (g)	Mass loss of polyamide (%)
50/50	5.00 ± 0.01	2.70 ± 0.04	93.20 ± 1.70



**FIGURE 1** Representative photomicrographs, both at 40× magnification, of a 50/50 polyamide/wool fiber mix (a) before CEW treatment and (b) after CEW treatment. The scale bar at the bottom right of each image depicts 100 μm [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com/doi/10.1002/app.51170)]

entirely to the dissolution and separation of polyamide, and that is also presented in the table as percent of the initial mass of polyamide in the mixture. In addition, representative photomicrographs of an initial fiber mixture, and of the wool sample after polyamide dissolution and separation are shown in Figure 1.

The two fiber types can easily be differentiated in the photomicrographs from their characteristic appearances. Wool exhibits distinctive scales and the polyamide appears as featureless cylindrical shapes. Both are evident in Figure 1(a). No evidence of intact polyamide fibers was observed after CEW treatment (Figure 1(b)), but also there was no evidence of damage to the wool fibers. The morphological appearance of wool fibers before and after CEW treatment is visually identical under the microscope.

The CEW treated fiber mixture also showed the presence of agglomerates or aggregates, which are the residues of reprecipitated polyamide. From the mass difference shown in Table 1, we estimate that ca. 7% by mass of the original polyamide content was retained in the wool after dissolution and separation of the 50/50 polyamide/wool mixture. As no polyamide fiber fragments were found, the retained polyamide mass is attributed to the polyamide solution that remained in the wool after filtration, from which the polyamide was regenerated as the wool fibers were rinsed with water.

### 3.2 | FTIR analysis

The FTIR-ATR absorption spectrum of the original PA66 fiber (trace A) and that after complexation with calcium

(trace B) are shown in Figure 2. For the latter, a mass of fibers was dissolved in the CEW mixture and left to rest in an uncovered vessel in the ambient atmosphere until all liquid evaporated. The FTIR-ATR measurement was performed on the solid residue.

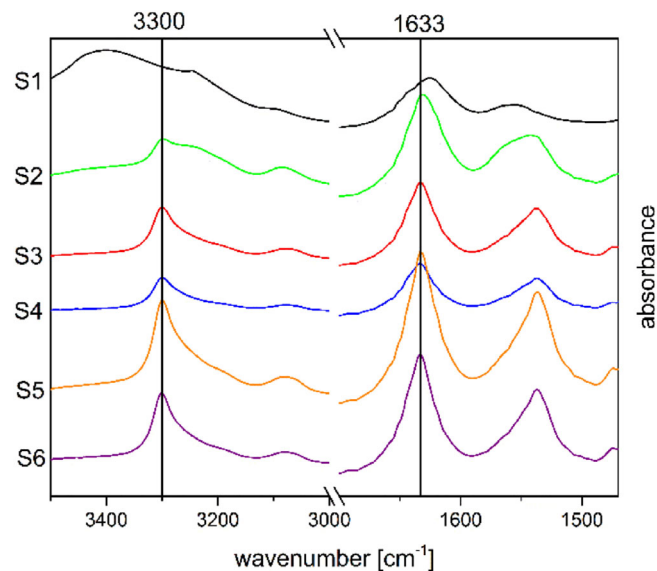
The mechanism of CEW action on polyamide is through the formation of Lewis acid-base complexes between the  $\text{Ca}^{2+}$  (Lewis acid) and the polyamide carbonyl group (Lewis base). Such complexation with a polyamide carbonyl, in addition to  $\text{Ca}^{2+}$ , has also been demonstrated with other Lewis acids (e.g.,  $\text{Li}^+$ ,  $\text{B}^+$ ,  $\text{Al}^{3+}$ , and  $\text{Ga}^{3+}$ ) on silk,<sup>24</sup> polyamide 6,<sup>25–27</sup> polyamide 66<sup>28–30</sup> and Kevlar.<sup>31</sup> The complexation at the carbonyl group disrupts the intermolecular hydrogen bonding ( $\text{N-H}\cdots\text{O}=\text{C}$ ) between polymer chains, leading to reduced crystallinity, improved interchain mobility, and ultimately to swelling and dissolution. It is also confirmed that the solvent does not cause any molecular degradation of PA, for example, chain scission or lowering the molecular weight.<sup>21</sup> In contrary, the complexation process is fully reversible and the reduction of  $\text{Ca}^{2+}$  concentration leads to decomplexation and reformation of hydrogen bonds (Figure 3).

The characteristic FTIR absorption bands of polyamide 66 and their assignments are listed in Table S1,<sup>32</sup> and they were observed in the original fibers (trace A, Figure 2). The polyamide Lewis acid complexation is generally reported to cause a blue shift of the Amide A peak (i.e., shift toward higher wavenumbers),<sup>29,33,34</sup> and a red shift of the Amide I peak (i.e., shift toward lower wavenumbers). The Amide A blue shift is attributed to the “free” N–H groups emerging as a result of the  $\text{N-H}\cdots\text{O}=\text{C}$  hydrogen bonds being disrupted, and





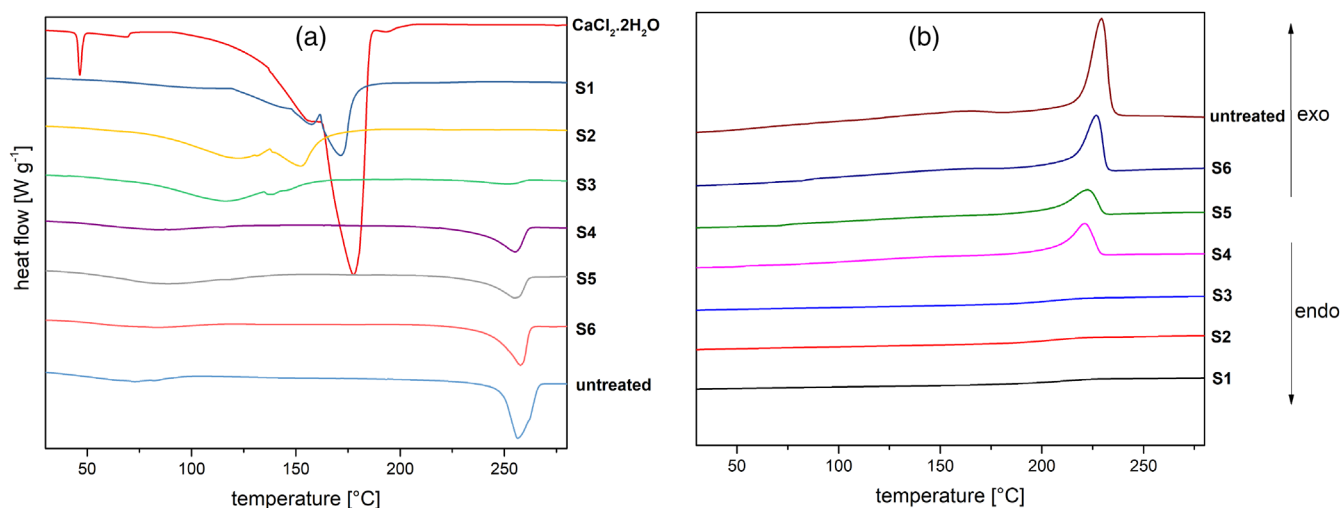
evinced no significant polymer melting peaks but exhibited broad endotherms in the 50–180°C range, which is attributed to the evaporation of calcium-associated water. The cooling thermograms of samples S1–S3 showed no evidence of crystallization exotherms, while samples S4–S6 showed exothermic crystallization peaks.



**FIGURE 4** FTIR-ATR spectra from the polyamide samples reprecipitated from their solutions in CEW by dilution with water, and rinsing for 0 min (S1), 5 min (S2), 10 min (S3), 20 min (S4), 30 min (S5), and 45 min (S6). For clarity, only the spectral regions about the amide I and amide II peaks are shown [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]

and these results suggest a level of calcium complexation in these samples that was sufficient to interfere with intermolecular hydrogen bonding and prevent crystallization. The heating thermograms of samples S4–S6 resembled those of the original fiber, with broad endotherms in the 50–100°C range that is associated with the evaporation of moisture adsorbed in the polymer, and endothermic melting peaks. However, the peak melting temperatures of these samples were lower than that of the original fiber. Similarly, the cooling thermograms of samples S4–S6 resembled those of the original fiber, with exothermic crystallization peaks, but with peak crystallization temperatures lower than that of the original fiber. In addition, the crystallization enthalpies in samples S4–S6 were generally lower than that of the original fiber. These results suggest the re-establishment of intermolecular hydrogen bonding in these samples to levels that were sufficient to allow for crystallization, but also that a residual level of calcium complexation was retained in the samples which accounts for the lowered crystallization temperatures and enthalpies.

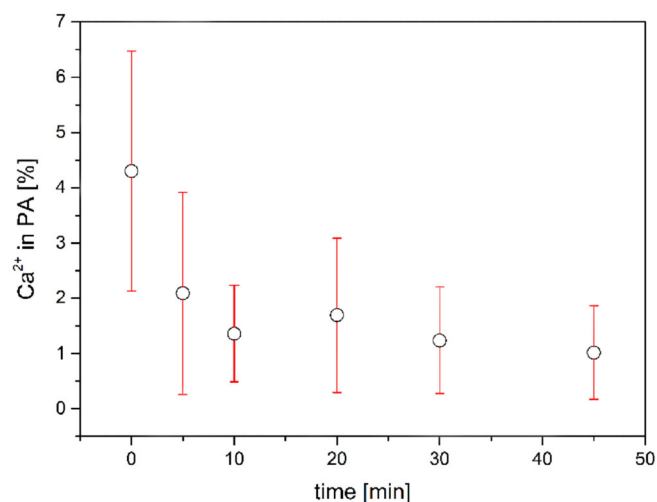
It may be of interest to note that in contrast to the DSC results, FTIR-ATR spectroscopy revealed no evidence of complexation in sample S3. The latter is a surface-limited technique with the depth of IR beam penetration into substrates of typically between 0.5 and 5  $\mu\text{m}$ ,<sup>35</sup> whereas DSC is a bulk-characterization method. Thus, it appears that in sample S3, there was decomplexation and re-establishment of hydrogen bonds at the surface of the reprecipitated solids but the overall



**FIGURE 5** Heat flow in DSC measurements of polyamide samples reprecipitated from their solutions in CEW by dilution with water, on (a) heating from 20 to 280°C and (b) cooling from 280 to 20°C and both at the rate of 10°C min<sup>-1</sup>. The sample designations S1–S6 are the same as in Figure 4. Both parts include results from the original fiber (untreated), and part (a) also includes the results from  $\text{CaCl}_2 \cdot 2\text{H}_2\text{O}$ . The results shown are representative examples from four measurements per sample [Color figure can be viewed at [wileyonlinelibrary.com](http://wileyonlinelibrary.com)]

**TABLE 2** The peak melting temperature ( $T_m$ ), peak crystallization temperature ( $T_c$ ), and crystallization enthalpies ( $\Delta H_c$ ) derived from DSC measurements. The values shown are the average  $\pm$  standard deviation from four replicate measurements

Sample	Rinsing period (min)	$T_m$ ( $^{\circ}\text{C}$ )	$T_c$ ( $^{\circ}\text{C}$ )	$\Delta H_c$ ( $\text{J g}^{-1}$ )
S1	0	–	–	–
S2	5	–	–	–
S3	10	$247 \pm 6$	–	–
S4	20	$255 \pm 4$	$224 \pm 12$	$40 \pm 17$
S5	30	$255 \pm 1$	$224 \pm 5$	$37 \pm 7$
S6	45	$256 \pm 2$	$228 \pm 6$	$55 \pm 13$
Untreated	–	$260 \pm 3$	$239 \pm 6$	$51 \pm 4$



**FIGURE 6** The calcium contents (wt%) determined with complexometric titration in samples S1–S6. The error bars represent the standard deviation [Color figure can be viewed at [wileyonlinelibrary.com](https://onlinelibrary.wiley.com)]

extent was not very significant. It also points to an “outside-in” mode of decomplexation, where the rate will depend on water diffusivity through the reprecipitating mass, and therefore may vary with the physical dimensions and any variations in local densities as the dissolved polymer falls out of the solution.

### 3.4 | Determination of calcium contents

The calcium contents in the reprecipitated polyamide as function of the rinsing period is shown in Figure 6. The values obtained with Equations (1) and (2) were converted to weight percent (wt%) units in constructing the plot. For reference, the calcium content in the original fiber was found to be  $0.004 \pm 0.001$  wt%.

After a rapid initial drop from ca 4.30 wt% (0 min rinse, sample S1) to 2.09 wt% (10 min rinse, sample S2),

the values did not vary by much. The samples rinsed for 10–45 min (i.e., S3–S6) exhibited calcium contents in the range of 1.02–1.70 wt%. One of the possible reasons for the very high standard deviation might be the inhomogeneity of the sample due to the accessibility of water into the PA solid phase during precipitation and wash-off processes as the accessibility most likely also changes during the decomplexation. The more solid decomplexed PA is formed at the outer, the higher is the barrier for water to access the core region of the sample. Furthermore, the lack of significant difference in calcium contents between sample S3 and samples S4–S6 was unexpected as we consider that in the DSC results, sample S3 showed no evidence of crystallization or significant melting, whereas both were observed in samples S4–S6. The crystallization behavior of PA (DSC measurement) seemed to be more sensitive to calcium content than IR investigation. Most likely, a relatively small number of disrupted hydrogen bonds was sufficient enough to disturb the ordering of PA chains and thus, the crystallization, but the number of “free” N–H groups was not enough to shift the Amide A peak to higher wavenumber (blue shift). However, an overall trend could be observed that the calcium content was decreased with increased rinsing time and the results also highlight the large difference in calcium content between sample S6 (rinsed for 45 min) and the original fiber, and indicate the need for either far longer rinses with water or a change of the rinsing medium to one containing an acid or chelating agent. More investigations on calcium decomplexation and wash-off are planned in future work to further clarify the mechanism.

## 4 | CONCLUSION

The work was dedicated to the investigation of the decomplexation process of precipitated polyamide from CEW mixture solution by a simple washing step and further built the basic understanding of the process for the selective separation through nondestructive dissolution

and recovery by reprecipitation and decomplexation and of PA from mixed textile waste and reuse of the PA fraction. There was a quantitative dissolution of the polyamide in the solvent by complexation with calcium ions, and it could be recovered from solution through decomplexation by the addition of water. In previous studies, we found the solvent does not affect the polymer molecular weight, that is, there is no significant degradation. It was also found that the decomplexation with water alone may not suffice to remove all calcium from the reprecipitated polyamide. In order to ensure an intake polymer structure for later processing, for example, fiber melt spinning, it is however importance to remove as much as possible of the residual calcium as it influences intermolecular interaction by disrupting the hydrogen bounds between the polymer chains. It may be necessary to follow the decomplexation by a treatment with chelating agent or mild acid.


These results suggest a potential advantage of selective nondestructive dissolution and recovery of PA fibers. However, many more questions remain to be answered, for example, how will process conditions, for example, temperature and agitation speed influence the dissolution (complexation) and recovery (decomplexation) efficacy, what will be the impact of colorants and other additives normally present in post-consumer textiles, what will be the melt-behavior and processability of recovered polyamide etc. The answers to these and other questions will be sought in ongoing and future investigations.

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## SUPPORTING INFORMATION

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