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Polyhydroxyalkanoate (PHA) purification through dilute aqueous ammonia digestion at elevated temperatures

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PHA (polyhydroxyalakanoates) are a family of microbial polyesters with the potential to replace polyethylene or polypropylene in many of their applications. These bioplastics are already in the market, but its presence is still limited, due to –among others- their high production costs or their final properties.¹ PHA recovery can have a big impact on both factors, as well as on the overall sustainability of the process.

Dilute ammonia digestion has been considered a promising method for PHA recovery, given the possibility of recycling NH₃ as a nitrogen source during the PHA production steps. However, most of the studies up to now had achieved low PHA purities and recoveries.² In the present study, we investigated how the digestion conditions affected the outcome of the purification, and proved that high PHA purity and recovery can be achieved at elevated temperatures (up to 90%). Moreover, PHA purified though NH₃ digestion was thermally stable during melting, with almost no reduction of the molar mass at this stage, revealing NH₃ digestion as a promising method for PHA recovery.

The experiments showed no purity increase from the initial material (64% PHA) and a low recovery (down to 68%) at temperatures below 75°C, regardless of the time of incubation and the NH₃ concentration. The trends were reversed when the temperature increased above 75 °C: more impurities were solubilized - attaining higher PHA purities (up to 90% at 140°C) - and PHA recovery improved (90%). The results revealed that less PHA monomers were
released at temperatures over 75 °C, which led to a higher degree of recovery, possibly due to changes in the polymer conformation at elevated temperatures.

On the other hand, severe molar mass reduction was observed at conditions maximizing the purity (140 °C). In this regard, sonication proved to be a valuable pre-treatment to enhance the PHA purity at conditions not resulting in severe molar mass reduction. A PHA purity of 86% and a PHA recovery of 92% was achieved at 115°C with a previous sonication pre-treatment (molar mass was 200 kg/mol). Despite the polymer was not absolutely pure, it was very stable during melting. It presented only a 10% reduction of the molar mass at 170 °C, comparable to chloroform extracted PHA. On the contrary, PHA purified with other digestion methods (such as H₂SO₄ or NaOH), with PHA purities close to 100%, presented a severe reduction of the molar mass during melting (80%).