Chemical recycling of End-of-Life wind turbine blades by solvolysis/HTL

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Abstract. The focus of this contribution is to highlight the challenges of chemical recycling of End-of-Life glass fiber composite (GFRP) waste from wind turbine blades utilizing solvolysis/HTL (hydrothermal liquefaction) methods based on subcritical water as solvent. A multitude of investigations have been published during the years regarding solvolysis of newly produced composite laminates and known thermoset composition (epoxy, polyester, and vinyl ester). However, a real wind turbine blade is more complex and constitutes of thermosets, thermoplastics, and other materials such as balsa wood. It is a very challenging task to separate these materials from each other within the wind turbine blade structure, so the premise for recycling is a mixed waste stream where little is known about the chemical composition. In the present study, the solvolysis process for GFRPs based on sub/supercritical water at 250-370 °C and 100-170 bar process conditions with catalyst (acid and base) and additives (alcohols and glycols) was studied and optimized. The samples used are representative for End-of-Life wind turbine blades. The aim is therefore to investigate if it is possible to develop a general process that can accept all material constituents in a real wind turbine blade, resulting in recycled glass fibers and a hydrocarbon fraction that can be used as a refinery feedstock.

1. Introduction
Wind power is an important source for production of renewable energy, and an increasing number of wind farms are built around the globe. However, the End-of-Life issues of glass fiber composite (GFRP) wind turbine blades are yet to be solved. Estimates have suggested that 330,000 t/year until 2028 and 418,000 t/year between 2028 and 2040 of composite materials from wind turbine blades will be deposited/incinerated worldwide [1]. Therefore, new innovative recycling processes for GFRP are needed. As the main driving force for recycling GFRP is the environmental impact rather than economic profitability, this question has been neglected until recently.

The focus of this work is to highlight the challenges of chemical recycling of composite waste materials from the wind turbine blades. The general structure of a wind turbine blade is based on both laminate and sandwich constructions in various parts, but the exact chemical constituents are very seldom disclosed. The wind turbine blade is made of glass fiber, thermosets (epoxy, polyester, and vinyl ester), thermoplastics [i.e. polyvinyl chloride (PVC), polyethylene terephthalate (PET) and polyurethane (PU)], balsa wood, adhesives and coatings [2]. This complex material mix leads to a great challenge for material recycling since the different materials are difficult to separate. For an economically viable process for chemical recycling of GFRPs, both major constituents of the blade (i.e. glass fiber and plastic) need to find usage in new future material streams i.e. recycled glass fiber and chemical building blocks.
One strategy is therefore to separate the insoluble GFRPs by utilizing a high temperature process based on sub/supercritical solvents i.e. solvolysis/HTL (hydrothermal liquefaction). At high temperatures and pressure (200-370 °C, 100-250 bar) water and alcohols can act as both solvent and reactant for GFRPs similarly to organic solvents. By hydrolysis reactions the plastic components are degraded to smaller fragments and the glass fibers are released from the composite network [3]. Solvolysis of GFRP with subcritical water results in glass fiber (solid material) and resin degradation products such as monomers, oligomers and polymers which separate into the oil or water phase depending on their polarity (Figure 1). The solvolysis also result in the formation of new repolymerized polymer structures i.e. oligomers or high molecular weight structures (char) [7].

Previous studies related to the use of chemical recycling by solvolysis of epoxy, polyester, and vinyl ester fiber reinforced composites (FRPs) can be found in the literature (reviews [3, 4]; water [5-7]; alcohols [4, 8]; glycols [9-14]; acetone [5]; acetic acid/hydrogen peroxide [15, 16]; wind turbine blade recycling [2]). Many of these studies focus on recycling of the more valuable carbon fiber composites (CFRP) due to higher market value of the recycled carbon fiber product vs. glass fiber. Another important issue is that most of the studies utilize newly produced composite laminates with one known thermoset and no blends of thermoset GFRPs. Therefore, it is difficult to translate the reported solvolysis studies to real samples from wind turbine blades where different optimized thermoset systems are used for distinct parts within the blade.

**Figure 1.** General flow diagram of solvolysis of wind turbine blade GFRP and the resulting product fractions of degradation in subcritical water.

A few studies have utilized material from wind turbine blades for chemical or thermal (pyrolysis) recycling [2, 17-19]. The focus of all studies was on polyester based GFRP. In the GENVIND project solvolysis with subcritical water at 300 °C and 100 bar was used for recycling GFRP from polyester wind blades into a solvolysis oil with high calorific value (40 MJ/kg). The recovered glass fibers were free of the resin and reported to have a tensile strength in the range of 80–90% of virgin fibers [2, 20]. Thermal recycling with a microwave pyrolysis (300-600 °C) on wind turbine blade waste based on polyester GFRP was studied by Åkesson et. al. [19]. Glass fiber with 75% strength of the virgin glass fibers could be recovered along with an oil with calorific value of about 36 MJ/kg. In another project EURECOMP, glass-fiber reinforced polyester was recycled by solvolysis based on hot water and high pressure. The process conditions were optimized at small scale reactor systems and upscaled to a large batch reactor system of 20 L. Solvolysis reaction at 270 °C led to 70 % in mechanical strength of the recovered glass fibers vs. virgin fibers [17].

Critical conclusions from literature available to date on chemical recycling of FRPs by solvolysis:

a. The optimization of reaction time and temperature for retaining highest possible strength of the recovered glass fibers leads to longer reaction times and a higher energy consumption. Increased reaction temperature yields significant reduction of the recovered glass fiber strengths [17].

b. High temperature water has been reported to cause detrimental chemical reactions with glass fiber, reducing their strength. Reports on solvolysis with organic solvents (i.e. alcohols and acetone) have claimed less damage on the recovered glass fibers and increased diffusion of solvent into the FRP due to changed hydrophobicity of the sub/supercritical media [4, 5, 8, 9].

c. Recovered glass fibers need to be washed with organic solvent (acetone) and resized before usage as reinforcement in secondary products [7, 17].

d. Polyester/vinyl thermosets are easier to degrade than epoxy based due to ester linkages can be hydrolyzed/trans esterified [3].
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- Heavily crosslinked matrix networks hinder the diffusion of solvent molecules and are therefore more difficult to degrade by chemical recycling. Sample size also affects the reaction rates i.e. grinded sample needs shorter time than larger chopped pieces [15, 16].

- Pretreatment with solvent (acetic acid/hydrogen peroxide) for swelling of the FRP can reduce the reaction time by increased diffusion of solvent into the composite structure [21].

The aim is therefore to investigate if it is possible to develop a general solvolysis/HTL process that can accept all material constituents in used wind turbine blades. Therefore, a hit-to-lead approach is adopted for depolymerizing End-of-Life GFRPs in subcritical water (250-370 °C, 100-170 bar) in small-scale equipment (7 mL), which enables screening of various reaction conditions. Experimental data from the solvolysis process of End-of-Life GFRPs and the resulting resin degradation products will be discussed.

2. Materials and experimental section

2.1. Materials
Two different blades with unknown material composition was used: glass fiber composite (GFRP, 1A, 1B, 1D, 2A, 2C and 3A), balsa wood (1C), polyester foam core (2B), rubber (3B) and PVC foam core (3C) (Figure 2 and 3). The different parts of the wind turbine blade were cut in pieces (approx. 10×10 cm) and were grinded to particles <1 mm size. In order to check the performance of the solvolysis equipment, a polyester based GFRP was used as reference sample in solvolysis experiments (PE).

Figure 2. Wind turbine blade 1 sample parts: glass fiber composite (GFRP, 1A, 1B, 1D, 2A and 2C), balsa wood (1C) and polyester foam core (2B) (Enercon).

Figure 3. Wind turbine blade 2 sample parts: glass fiber composite (GFRP, 3A), rubber (3B) and PVC foam core (3C) (blade type LM 29.0 An Bonus 1.3 MW) (Anmet)
2.2. Solvolysis

Solvolysis reactions were performed in sealed high-pressure vessels (10 mm inner diameter, stainless steel) with an internal volume of 7 mL, connected to a safety pressure valve (180 bar). In a typical solvolysis experiment 0.3-0.5 g of sample was heated with 5 mL of liquid in sealed pressure vessels to temperatures between 230-370°C in a tube furnace. After the reaction, the pressure vessels were cooled with water to room temperature and the solvate was rinsed with water, acetone or alcohol and the solid material were separated from the liquids by filtration. The product oil was isolated by extraction with ethyl acetate and acetone, followed by rotary evaporation. Due to the low sample size the oil and the water phases did not separate without the addition of extraction solvent unless at minimum of 5 experiments were combined. The resin degradation yield was calculated by equation (1) using the resin content in the sample before and after solvolysis equation (1).

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\text{Equation (1)} \quad \text{Resin degradation yield} \% = \frac{W_1 - W_2}{W_0} \times 100
\]

\(W_1\) – weight of composite before solvolysis
\(W_2\) – weight of solid material after solvolysis
\(W_0\) – weight of epoxy resin in composite before solvolysis

Unfortunately, the yield calculated on the weight of solid material before and after solvolysis resulted in a high uncertainty since the sample parts are often deposited in the reaction vessel. Instead, the fraction of epoxy resin in the sample before and after solvolysis, determined by TGA, was used to calculate \(W_0\) and \(W_2\) based on the sample weight \(W_1\). For plastics without glass fibers, the yield was calculated on the weight of solid material before and after solvolysis using the average results of triplicate experiments due to the high result variation.

2.3. Chemical characterization

The TGA analysis were performed on a Mettler Toledo Star System. The samples (10 mg) were heated in ceramic pans from 50°C to 600°C under \(\text{N}_2\) at a heating rate of 10 K/min. Non-homogenous samples were grinded before analysis. The GC-MS analyses were performed on an Agilent 6890N gas chromatograph coupled with an Agilent Model 5975 Inert XL Mass Selective Detector, equipped with a DB-5MS column (30 m × 0.25 mm ID, 0.25 μm film thickness), using helium as carrier gas at 1.0 ml/min and a EI detector of 70 eV. Compounds were identified by NIST 08 library of mass spectra. The Fourier Transform Infrared (FTIR) spectra were recorded on a NICOLET 6700 apparatus (Thermo Electron Corporation, USA) equipped with a Smart Orbit ATR (Attenuated Total Reflection).

3. Result and discussions

After screening a multitude of different solvolysis reactions with different catalyst and co-solvents such as acids, bases, organic solvents, peroxides, surfactants and catalysts (unpublished results), the most promising solvolysis reaction conditions for degradation of GFRP appears to be ethylene glycol, propylene glycol or water combined with alcohols and catalyzed with base at temperatures of 270-330°C. The solvolysis efficiency of degrading the organic material (thermoset and thermoplastics) from the GFRP blade parts are presented in Table 1 and 2.
Table 1. Resin degradation efficiency in solvolysis of GFRP wind turbine blade parts (1A, 1B, 1D, 2A and 2C), foam core (2B) and polyester GFRP reference (PE) from different reaction conditions.

| Solvolysis experiment                  | 1A (%) | 1B (%) | 1D (%) | 2A (%) | 2B (%) | 2C (%) | PE (%) |
|----------------------------------------|--------|--------|--------|--------|--------|--------|--------|
| Ethylene glycol, 270 °C, 16h           | 74     | 85     | 50     | 75     | >90    | 60     | >90    |
| Propylene glycol, 270 °C, 16h          | 52     | 80     | 40     | 85     | -      | 55     | 90     |
| Water, 1-propanol (20%) and KOH (10%), 330 °C, 4-16h | 15-55 | 10-50 | -      | 10-64 | 88     | -      | 85     |
| Water, 330 °C, 6h                       | <10    | -      | -      | <10    | -      | -      | -      |
| Resin content of starting material b    | 70     | 35     | 46     | 46     | -      | 37     | 20     |

a Yield after 4h; b Resin content determined by TGA.

Table 2. Comparison of the solvolysis reaction times and temperatures effects of degradation efficiency in water, 1-propanol (20%) and potassium hydroxide (10%) for GFRP sample 1A.

| Experiment | Sample (°C) | Time (h) | Resin degradation (%) |
|------------|-------------|----------|-----------------------|
| 1          | 270         | 2        | < 10                  |
| 1          | 270         | 16       | 15                    |
| 2          | 330         | 2        | 20                    |
| 2          | 330         | 6        | 40                    |
| 2          | 330         | 16       | 55                    |

3.1. Solvolysis comparison between water and ethylene/propylene glycol-based systems of GFRPs

Subcritical solvolysis of the epoxy-based samples 1A and 2A in water without additives resulted in poor yields (Table 1). During the optimization of the solvolysis, the use of glycols, 1-propanol and KOH turned out to be the best ways to improve the degradation efficiency. Ethylene glycol and propylene glycol at 270 °C were found to work more efficiently for the solvolysis wind turbine blade 1 (1A, 1B, 1D, 2A and 2C) yield 50-85% resp. 40-85%, Table 1) compared to water based solvolysis (yield 10-55%, Table 1). However, when the recovered glass fiber fraction from solvolysis of 1A in water/1-propanol/KOH for 4 hours was further extracted in acetone for five days in room temperature, the resin was fully removed from the glass fiber. This preliminary result indicates that major parts of thermoset resin in 1A is degraded in water/1-propanol/KOH at 330 °C, forming a solid organic residue on the glass fiber. This suggests that the epoxy thermoset is degraded to a thermoplastic that can be removed from the glass fiber by extraction in organic solvents, and that the epoxy resins are degraded to a higher degree than the yields presented in Table 1 for solvolysis of GFRP in water/1-propanol/KOH at 330 °C. The use of organic solvents such as acetone, alcohols and glycols has a great impact on the sustainability of the solvolysis. Further work is needed to determine the most economical and sustainable solvolysis system for GFRPs.
Comparison of the degradation yields of sample 1A and 1B (70% and 35% resin content respectively) indicates that the resin vs. glass fiber content has minor importance for degradation efficiency.

3.2. Solvolysis and influence of reaction time and temperature of GFRPs
In general, the most effective solvolysis is achieved when the reaction time is prolonged overnight (16 h), while reactions at higher temperatures and shorter reaction time do not result in higher yields. A study on the degradation efficiency of sample 1A with varied temperatures and reaction times in water 1-propanol and base illustrates this relationship (Table 2). The solvolysis performs poorly at 270 °C (yield <10-15%) and gives low yields at 330 °C with short reaction time (20-40%, 2-6 h) and after reaction overnight at 330 °C finally yields 55% removal of the thermoset.

3.3. Solvolysis of polyester- and epoxy-based GFRPs
The system investigated in the present contribution was wind turbine blade 1 (Figure 2) with unknown chemical composition of all ingoing materials. Therefore, a polyester based GFRP was used in the solvolysis for comparison (PE, Table 1). The polyester sample yielded higher degradation yields than the wind turbine blade 1 samples (Table 1). These yields are in good agreement with observations from other studies on polyester GFRPs and illustrate the large difference in resistance to solvolytic degradation between polyester- and epoxy-based composites [3].

3.4. Chemical analysis of solvolysis fractions from GFRPs degradation of wind turbine blade 1
Wind turbine blades’ chemical compositions are well-kept trade secrets. Therefore, the chemical contents of the plastics were characterized by FTIR and GC-MS of the starting materials as well as of the solvolysis products. The GFRP thermosets give poor signals and require long scanning times (high glass fiber content up to 65%). However, the GFRP samples from wind turbine blade 1 are identified as epoxy resins according to FTIR signals and bisphenol A detected by GC-MS in the solvolysis extracts. The foam core 2B is identified as polyester both by FTIR and GC-MS of the solvolysis extracts.

Chromatographic analysis of the oil phase fractions with GC-MS generally reveal low content of monomeric organic compounds (generally < 1% of the oil mass) which indicates that the polymers break down into larger oligomeric fragments or hydrophilic compounds that end up in the aqueous phase. Therefore, the GC-MS analysis only reveals a small part of the reaction products. Elemental analysis (CHN) and characterization of ground elements with X-ray fluorescence (XRF) are important tools for identifying starting materials and oil phase composition. For the GFRP samples (Figure 2 and 3), calcium and silicon are the main elements due to glass fiber content, with traces of chlorine (≤0.2%) and iron (≤0.8%), while the hydrocarbon content varies between 30-50%. CHN analysis of samples 1A and 1B showed nitrogen content (2.7 resp. 2.4%) indicating that these GFRPs contain amine cured epoxy thermosets. The corresponding solvolysis oil/wax products showed less than 0.1% of nitrogen, silicon, chlorine and iron. This indicates that the solvolysis can be a future tool for separating unwanted additives such as nitrogen-containing polar compounds and inorganic ions usually found in the wind turbine blades. For further upgrading and use of the produced solvolysis oil into resins, chemicals or fuels, the removal of chlorine and nitrogen atoms is essential. This study indicates that solvolysis have the possibility to act as a recycling method for cleaning mixed plastic waste streams like GFRPs from wind turbine blades before further processing and upgrading in a refinery [22].

3.5. Solvolysis and chemical analysis of thermoplastics and balsa wood parts from wind turbine blades
The wind turbine blades sandwich structures generally contain a foam core composed of other plastics such as PVC, polyester and PU or balsa wood. It seems possible to separate these core materials from the GFRP at an early stage of the solvolysis. Foam core samples 2B and 3C are nearly fully degraded in water/1-propanol/KOH at 330 °C after 4 h. These reaction conditions result in ≤15% degradation of the
epoxy based GFRP samples. The foam core 3C was found to have a chlorine content of >10% according to XRF, indicating PVC as the main component. Preliminary results from the XRF of the resulting oil phase fraction show only 2% chlorine which suggests that the chlorine content can be reduced from polymeric materials by solvolysis. Polymers containing chlorine are problematic in all types of chemical recycling due to formation of strong acids at high temperatures (i.e. hydrochloric acid, HCl) [23]. Therefore, this solvolysis technique seems to be a promising tool for removal of unwanted chlorine atoms prior to use in industrial chemical processes or combustion facilities.

Balsa wood core material was also degraded by solvolysis in water/1-propanol/KOH at 330 °C and yielded a fraction that shows the characteristics of paper pulp. These reaction conditions are similar to those used by the pulp and paper industry in existing plants (Kraft process) to produce cellulose pulp [24, 25]. The degraded balsa wood fraction ends up in the filtrated solid materials after solvolysis and is possible to separate from the glass fibers for small scale experiments.

3.6. Solvolysis experiment in larger scale of wind turbine blade 1
In order to examine the solvolysis efficiency on larger samples, a part of wind turbine blade 1 was sawn to pieces of 5 x 35 x 4 mm (0.5 g), displayed in Figure 4. A two-step process with 16 hours in ethylene glycol at 270 °C followed by 3 hours in water with 1-propanol (20%) and KOH (10%) at 330 °C yields 75% resin removal. The recovered glass fiber fraction from the first step of the treatment yields a material that can easily be cut into smaller fragments. In a larger process set up this could be a way forward to reduce time and cost for shredding or grinding of the GFRPs which is resource consuming.

![Figure 4. Solvolysis of a larger piece GFRP (5x35x4 mm, left) of wind turbine blade 1 in two reaction steps: 1. ethylene glycol (270 °C, 16 h) 2. Water,1-propanol (20%) and KOH (10%) at 330 °C for 3h. Recovered glass fiber fraction from solvolysis to the right in the figure.](image-url)

4. Conclusions
A chemical recycling method for samples from decommissioned wind turbine blades through solvolysis have been investigated and optimized to be able to separate plastic resins from glass fibers in a resource efficient approach. Most of the sample resins are characterized as epoxy which are difficult to degrade and separate from the glass fiber without the use organic solvents in addition to long reaction times. The best reaction conditions are achieved in a two-step process where the GFRP material is heated to 270 °C in ethylene glycol for 16 hours followed by heating to 330 °C in water/1-propanol/KOH for 3 hours.

Preliminary analysis of the produced oil phase products from wind turbine blade GFRP and foam cores indicate removal of nitrogen-containing hardeners in epoxy and a reduction of the chlorine content from PVC-based foam cores. Producing solvolysis oil with low levels of nitrogen and chlorine is important for further upgrading of the solvolysis oil to useful end products i.e. resins, chemicals or fuels. This study indicates that solvolysis have the possibility to act as a recycling method for cleaning mixed...
plastic waste streams like GFRPs from wind turbine blades before further processing and upgrading in a refinery.

However, large challenges still persist regarding chemical recycling with solvolysis of mixed waste streams of End-of-Life GFRPs and requires continued method development in order to reduce the consumption of energy and chemicals. One of the major challenges are the long reaction times due to thick and dense materials, and future work will focus on the use of flow reactors and recycled solvents for the optimization of the solvolysis.

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