Recyclable epoxy systems for rotor blades

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Abstract. The global energy demand is largely met by power generated from fossil fuels (Thermal Power plants) and nuclear means (Nuclear Power plants). Even though both these technologies have been used for decades, they have huge environmental impact and severe sustainability issues with depleting fossil fuel and serious concerns about the spent nuclear rods disposal. With growing awareness of impact on the environment and finding a sustainable solution for power generation, wind energy offers a lasting solution that addresses both issues, however it is still not truly sustainable as the primary materials used for manufacturing of rotor blades are non-recyclable. A new revolutionary technological solution has been invented where in, the epoxy thermoset can be recovered as thermoplastic and the reinforcement in composite can be re-used, making it a first of its kind in the thermoset industry segment. The Recyclable Epoxy Systems for rotor blades provide a path breaking solution that enables recycling of epoxy thermoset, recovery and re-use of the reinforcement and the matrix in a fibre reinforced composite. Leveraging from the proprietary Recyclamine® platform technology, series of recyclable epoxy systems are developed for wet lay-up and infusion processes to meet the requirements for both on-shore and off-shore rotor blades, including next generation rotor blades. The systems are characterized by determining process and performance properties and found to provide distinct advantages in comparison to the conventional non-recyclable epoxy systems. The recycling of composites made from recyclable epoxy systems is successfully demonstrated by low energy solvolysis followed by recovery of the reinforcement and epoxy matrix as thermoplastic. The recovered reinforcement and epoxy thermoplastic are re-used to make composite and representative thermoplastic object respectively, deriving value from the waste and closing the loop to make the wind energy industry sustainable for a circular economy.

1. Background
Wind Energy as a renewable source of energy has come a long way in being recognized as a major alternative source to the conventional energy sources based on fossil fuels. The global cumulative installed capacity of wind power generators reached 651GW at the end of 2019 witnessing a 10 % increase over the previous year [1]. The growth and maturation of the wind industry is duly supported by technological advancements in wind turbine rotor blades. The emergence of new – longer, stiffer and aerodynamically advanced rotor blade designs has enabled to harness more energy from the wind.
Designers and rotor blade manufacturers are continuously looking for new performance driven, robust materials to support the latest and future developments.

Incidentally, the industry is also facing compelling issues, such as process waste management and end of life management of aged rotor blades. It is estimated that 85% of the components of a wind turbine can be recycled and re-used except the rotor blades due to the non-recyclable thermoset matrix [2]. In addition it is estimated that 10% of the process waste generated during manufacturing of a rotor blade is non-recyclable. The present methods of waste management (Figure 1) such as landfills, pyrolysis, incineration, and pelletization are either not environment friendly or are energy intensive and fail to derive value. Solvolysis, involving nucleophilic substitution or elimination, is a clean and efficient method, however its use is limited due to non-recyclable nature of the epoxy thermoset.

![Figure 1: Present Methods for End of Life Waste Management of Rotor Blades](image)

With >4000 aged rotor blades scheduled to be decommissioned annually [2] and lack of full-proof or robust methods to manage waste, the growth and sustenance of wind energy is at stake.

2. Recyclamine® : Concept and Technology
Epoxy resins are preferred as polymer matrix materials for manufacturing of rotor blades on account of their versatility and outstanding process & performance properties. The epoxy resin systems used as matrix for manufacturing of rotor blades comprise of epoxy resin component and curing agent or hardener component which react to cross link and cure into rigid three dimensional infusible network that cannot be re-formed, re-used or re-cycled. With emergence of new rotor blade designs, epoxy resin systems are being continuously developed to align with rotor blade manufacturer and the designer’s requirement, however the non-recyclable nature of epoxy thermosets does not address the compelling issue of process waste management and end of life management of rotor blades.

Reculamine® technology provides an opportunity with path breaking and lasting solution that enables recyclable epoxy thermosets [3][4]. The technology is based on specifically engineered recyclamine® curing agents or hardeners which cross link with epoxy resins to provide a network with cleavage points. The matrix comprising of epoxy resin and recyclamine hardeners in polymer composites, can be cleaved by solvolysis under specific conditions, leading to recovery of reinforcement such as carbon fiber, glass fiber, kevlar® and the epoxy matrix as recyclable thermoplastic. The
recovered reinforcement and epoxy thermoplastic can be re-used and re-purposed. The concept is depicted pictorially in Figure 2.

![Figure 2: Schematic depicting Curing of Epoxy Resin Systems (Non-Recyclable v/s Recyclable)](image)

The Recyclamine® technology is a platform chemistry with series and wide spectrum of curing agents providing fast to slow reactivity & latency, low to high temperature resistance, chemical resistance and recyclability (Figure 3).

![Figure 3: The Recyclamine® Curing Agent Platform](image)

### 3. Recyclable Epoxy Systems from Recyclamine® Technology:

The Recyclamine® technology was leveraged for the development of recyclable epoxy resin systems for rotor blades. Laminating resin systems for wet lay-up process and infusion process were developed by selecting suitable epoxy resins [5] and appropriate Recyclamine® hardeners. Series of recyclable systems with varying reactivity were developed and characterized for process and performance properties (Table 1).
Table 1: Physical, Process and Mechanical Properties of Recyclable Epoxy Systems

- Process and Performance Characterization of Recyclable Epoxy Infusion System E

The properties of recyclable epoxy systems were found to be comparable to the conventional wet lay-up and infusion systems used for rotor blades manufacturing. In order to illustrate advantages of recyclable systems, infusion System E is selected as a representative example in this paper. The system E was specifically developed considering the requirements of offshore blades which are large structures typically > 70 meter in length and necessitate slow reacting matrix systems to ensure controlled and defect free infusion of the reinforcement during processing.

The recyclable resin system E was designed as an extension of the proprietary ultra-slow fast cure technology from Aditya Birla Chemicals [8]. The resin component of the system was formulated by selecting epoxy resins that could enable low viscosity and provide ultra-slow reactivity with the hardener component based on appropriate Recyclamine® molecules. The system was characterized for process and performance properties in comparison to conventional slow reacting non-recyclable system used for offshore blades.

4.1. Processing Properties of Conventional and Recyclable System E

The processing properties of the conventional and recyclable system were determined under identical conditions (temperature, humidity, process and test method) and results were compared (Table 2).
Table 2: Processing Properties of Conventional and Recyclable System E

The recyclable epoxy system E provided lower initial mix viscosity and significantly slower rise in viscosity, longer pot life compared to the conventional system. These properties are indicative of improved processability, faster and controlled infusion of the reinforcement leading to defect reduction in rotor blade manufacturing. Importantly the glass transition temperature (Tg) which relates to degree of cross-linking was found to be comparable for the conventional and recyclable system E, which indicates that the slow reactivity of recyclable system will not have an influence on in-mold time thus the cycle time to manufacture rotor blade will be unchanged.

4.2. Performance Properties of Conventional and Recyclable System E

The performance properties of conventional and recyclable system were determined by mechanical properties, fatigue behavior under identical conditions (temperature, humidity, test method and equipment) and results were compared.

Table 3: Comparative Mechanical Properties of Cured Epoxy Matrix

The tensile and flexural properties, strength and stiffness of the cured epoxy matrix for 10 specimens per series were found to be considerably higher for the recyclable system E compared to the conventional system (Table 3). The characterization of mechanical properties for glass reinforced epoxy composite
was done by preparing laminates by vacuum assisted resin transfer molding (VARTM) process using uni-directional (1200 gsm) and bi-directional (800 gsm) fabrics with fiber volume fraction, 50-55%. Tensile, shear and fracture toughness (G1c) properties were determined, results indicated higher values for the recyclable system E compared to the conventional system (Table 4).

| Property | Unit | Conventional System | Recyclable System E |
|----------|------|----------------------|---------------------|
| Tensile strength | MPa | 815.64 | 866.48 |
| Fracture strain | % | 2.02 | 2.00 |
| Modulus (Chord 0.05%-0.25%) | MPa | 43,507 | 44,355 |
| Shear stress at 5% shear strain | MPa | 39.11 | 43.47 |
| Shear modulus | MPa | 3,220 | 3,555 |
| Tensile strength | MPa | 124.39 | 136.20 |

Table 4: Comparative Mechanical Properties of Glass Reinforced Epoxy Composite

The fatigue behavior of conventional and recyclable system was determined by conducting test for reinforced epoxy laminate made from uni-directional glass fabrics. Test was conducted in strain control mode, at 40% of ultimate strain and ratio R=0.1, frequency = 4 hertz. Change in load with number of cycles was monitored, test was continued until 800,000 cycles. Residual tensile properties; strength, elongation and E-modulus, of specimens were determined and compared with specimens before fatigue (Figure 4).

Figure 4: Comparative Tensile Properties, before and after fatigue test
5. Recycling of Glass Reinforced Epoxy Composite and Recovery of Matrix and Reinforcement

The recycling of glass reinforced epoxy composite made from recyclable epoxy system E and recovery of matrix and reinforcement was demonstrated by preparing a laminate with VARTM process using tri-axial fabrics (1200 gsm). The laminate was trimmed and subjected to solvolysis by immersing in recovery solution and conditioned at 80°C for 6 hours to enable cleavage of the recyclable epoxy matrix and its dissolution in the recovery solution. The glass fabric reinforcement of the composite, recovered from the recycling process was rinsed and dried. Recovery solution containing the dissolved epoxy matrix was filtered, neutralized and coagulated to recover the epoxy matrix as thermoplastic (Figure 5).

![Image of recycling process](image)

**Figure 5:** Recycling of Composite and Recovery of Matrix and Reinforcement

6. Re-use of Recovered Reinforcement and Thermoplastic

The tri-axial reinforcement recovered from recycling process was re-used by preparing laminate by VARTM process using recyclable epoxy system E as the matrix. Laminate was also prepared with fresh tri-axial (1200 gsm) reinforcement keeping all parameters identical. Specimens were extracted from both laminates and characterized for tensile properties using Universal testing machine Instron 5569 and clip on extensometer. Results indicated 10% lower tensile strength and modulus for the laminate prepared from recovered reinforcement evidently due to distortion in fibre alignment, however the tensile strain was found to comparable to the laminate prepared from fresh reinforcement (Figure 6).
Figure 6: Comparative Tensile Properties of Laminate from Fresh and Recovered Fabrics

The epoxy thermoplastic recovered from recycling process was dried and characterized for physical, thermal and mechanical properties such as melt flow index, glass transition temperature, tensile and flexural properties (Table 5).

| Property                           | Test Standard     | Unit       | Results |
|------------------------------------|-------------------|------------|---------|
| Melt Flow Index (190°C @ 2.16 kg) | ASTM D 1238 [15]  | minutes    | 10      |
| Specific gravity                   | ASTM D 792 [16]   | gm/cm³     | 1.19    |
| Glass Transition Temperature (Tg)  | ASTM D 3418 [17]  | °C         | 78      |
| Tensile Strength                   |                   | MPa        | 51.2    |
| Tensile Modulus                    | ASTM D 638 [18]   | GPa        | 2.90    |
| Elongation at break                |                   | %          | 30-110  |
| Flexural Strength                  | ASTM D 790 [19]   | MPa        | 100     |
| Flexural Modulus                   |                   | GPa        | 2.7     |

Table 5: Characterization of Recovered Thermoplastic
The thermoplastic was compounded with 80% polyethylene by weight and injection molded to re-purpose and make foot strap insert used in surfboards (Figure 7).

![Figure 7: Foot Strap Insert from Recovered Thermoplastic](image)

### 7. Summary and Conclusion

The Recyclamine® technology enables recycling of epoxy thermosets and further recovery, re-use of reinforcement, epoxy matrix in fibre reinforced composite. The technology was successfully leveraged to develop series of recyclable epoxy resin systems for rotor blades thereby providing path breaking and lasting solution to the compelling issue of waste management faced by the wind industry.

The recyclable epoxy systems were characterized for process and performance properties and benchmarked with conventional, non-recyclable epoxy systems. The recyclable epoxy infusion system E, developed for longer, aerodynamic offshore blades provided low viscosity and ultra-slow reactivity which is advantageous for improved impregnation and process defect reduction and importantly cured in the same time as the conventional system. Composite laminate prepared from the system was recycled by solvolysis to recover reinforcement and epoxy matrix as thermoplastic. Re-use of the reinforcement and re-purposing of the recovered thermoplastic was demonstrated to close the loop to make wind energy “Sustainable for a Circular Economy”.

### 8. References

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