RADICAL INDUCED CATIONIC FRONTAL POLYMERIZATION OF EPOXY SYSTEM

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INTRODUCTION

Radical induced cationic frontal polymerization (RICFP) is an elegant technique to cure epoxy resin. Combining advantages of thermal curing (bulk curing) and photopolymerization (fast curing, low energy consumption, and long pot-life) make the RICFP beneficial in industrial applications.[1] A formulation for RICFP consists of an epoxy monomer, e.g. bisphenol-A-diglycidylether (BADGE), tetraphenyl-1,2-ethanediol (TPED) as a radical thermal initiator, and an antimonite (I-Sb) or aluminate (I-Al) based diaryl iodonium salts as a photoacid generator. The presence of the two initiators allows the RICFP to be initiated by UV light or a thermal stimulus. The so liberated acid initiates a cationic ring opening polymerization of the epoxy rings of the resin. This exothermic reaction generates the heat necessary to cleave the radical thermal initiator, which provides radicals that can undergo a redox reaction with the photoacid generator causing again acid liberation (Figure 1).[2,3]

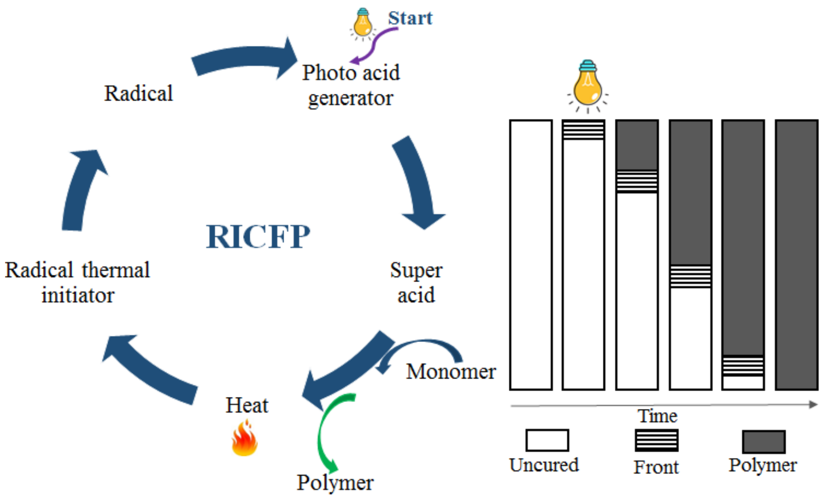

Figure 1: Mechanism and scheme of RICFP

With these advantages, the RICFP deserves to be deeply investigated for composite manufacturing which is commonly performed at elevated temperature, for many hours. Furthermore, the RICFP is a promising technique for applications like chemical anchor bolts (Figure 2) and under water curing which were difficult to carry out with conventional techniques until now.

EXPERIMENTS

The RICFP was conducted in a PTFE mold. An UV/Vis-light of 320 to 500 nm and a light intensity of 3 W/cm^2 at tip of a light guide were used to initiate the frontal polymerization. After initiation, the reaction will propagate without further irradiation being necessary.

RICFP under water: the standard formulation for RICFP contains BADGE, TPED as the thermal radical initiator, and I-Al as the photoacid generator. To test the possibility to conduct the RICFP under water, process of filling the mold with the formulation and irradiation step were done underwater.

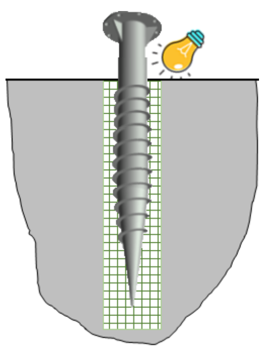

Figure 2: Chemical anchor bolts RICFP experiment

Chemical anchor bolt: this is a way to fix screws or bolts in walls without the need of common anchor bolts. In a common brick, holes of 5 mm diameter and 30 mm depth were drilled. Then, the prepared solution was filled in the drilling holes and a screw with 3 mm diameter was positioned in the center of the hole. Excessive formulation was removed before the initiation of the RICFP (Figure 2).

RICFP for epoxy composites: various fillers and fibers were used. Due to high viscosity and low reactivity of the BADGE, some reactive diluents were tested. The filler and fiber were used from low to high content to investigate the RICFP.

RESULTS AND DISCUSSION

RICFP underwater: the experiment indicated that the RICFP was successfully conducted under water (Figure 3). This result can be used for applications that had been very difficult to carry out until now, such as filling underwater cracks in bridge pillars or dams, or repairing pipes during ongoing operation.[4]

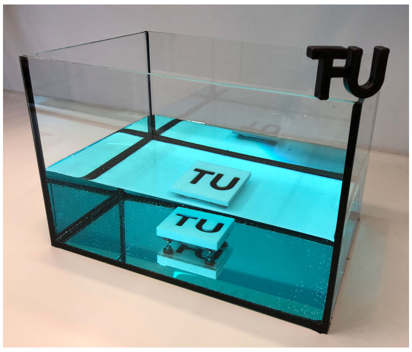

Figure 3: RICFP under water experiment

Chemical anchor bolts: the whole polymerization of the anchor bolt was done in a very short period of time by the RICFP. The stability of the specimen were tested by pull-out resistance. The result illustrated that the anchor bolt can resist the pull-out force up to 1000 N.

The RICFP for composite was successfully conducted with various fillers, for instance aluminum, milled carbon fiber, glass microspheres, aluminum nitride, silicon carbide, copper, graphite, mica, and carbon nano tubes (Figure 4). Furthermore, formulation containing reactive diluent can be used to produce composites with high filler content up to 75 vol% glass microspheres, 40 vol% milled carbon fiber, and 30 vol% aluminum nitride. The RICFP for fiber reinforced epoxy composite was investigated with carbon fiber and glass fiber.[4] 35 vol% of each fiber can be added in the specimen. Tensile testing revealed that the tensile strength, Young’s modulus, and elongation at break of the composite prepared by RICFP were held at the same level of those value of the specimen cured by the conventional technique.

Figure 4: Composite specimens

CONCLUSION

The RICFP can be conducted under water. The mechanically stable anchor bolt were successfully prepared by RICFP. The RICFP for composite was not only successfully conducted with variety of fillers and fibers, but also very high filler content.

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