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Effects of Sulphuric Acid Concentrations during Solvolysis Process of Carbon Fiber Reinforced Epoxy Composite

(Kesan Kepekatan Asid Sulfurik semasa Proses Solvolisis Komposit Epoksi yang Diperkuat oleh Gentian Karbon)

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ABSTRACT

Developing a cost-effective technique of reclaiming carbon fibers without significantly deteriorating their quality has gained much importance. This paper reports on reclaiming carbon fibers from the carbon fiber reinforced polymer (CFRP) by solvolysis technique using sulphuric acid. The main objective of this work was to determine the lowest sulphuric acid concentration which can remove the epoxy resin from the composites, at room temperature and atmospheric pressure. The sulphuric acid concentrations used ranged from 11 to 18 mol L⁻¹ with 30 min reaction time and 50 mL volume. Thermogravimetric analysis (TGA) showed that the fiber content of the composites is 68% by weight. From the solvolysis process, mass of decomposed epoxy resin decreased with decreasing acid concentrations. SEM images show that the residual epoxy adhering to the fibers after solvolysis increased with decreasing molarity. Acid concentrations lower than 15 M were found to be not effective in removing the epoxy resin from the carbon fibers. The tensile strength and Young's modulus of all the reclaimed fibers is marginally lower than the untreated carbon fibers. The tensile strength was found to increase as the acid concentration decreases with fibers in 15 M acid showed the highest tensile strength of 3.43 GPa (99.1% strength retention). Similar trend was also observed for Young's modulus. The study shows the potential of sulphuric acid to reclaim carbon fiber from the CFRP with 15 M giving the optimum properties in terms of tensile properties retention and epoxy resin decomposition.

Keywords: Carbon fiber reinforced polymer; epoxy resin; solvolysis; sulphuric acid; tensile properties

ABSTRAK

Penghasilan teknik yang kos efektif untuk memperoleh gentian karbon tanpa mengurangkan kualiti secara ketara telah menjadi semakin penting. Kertas ini melaporkan tentang perolehan gentian karbon daripada gentian karbon diperkuatkan dengan polimer (CFRP) melalui teknik solvolisis menggunakan asid sulfurik. Objektif utama kerja ini adalah untuk menentukan kepekatan asid sulfurik terendah yang boleh menguraikan resin epoksi daripada komposit pada suhu bilik dan tekanan atmosfera. Kepekatan asid sulfurik yang digunakan dalam julat antara 11 dan 18 mol L⁻¹ dengan masa tindak balas 30 min dan isi padu 50 mL. Analisis Termogravimetrik (TGA) menunjukkan bahawa kandungan gentian adalah 68% daripada berat komposit. Daripada proses solvolisis, jisim resin epoksi yang terurai berkurangan dengan berkurangnya kepekatan asid. Imej SEM menunjukkan baki epoksi yang melekat pada gentian selepas solvolisis bertambah dengan berkurangnya molariti. Pada kepekatan asid kurang daripada 15 M, didapati tidak berkesan dalam menguraikan resin epoksi daripada gentian karbon. Kekuatan tegangan dan modulus Young untuk semua gentian yang diperolehi adalah sedikit rendah daripada gentian karbon yang tidak dirawat. Kekuatan tegangan menunjukkan peningkatan apabila kepekatan asid berkurang dengan gentian dalam asid 15 M menunjukkan kekuatan tegangan tertinggi iaitu 3.43 GPa (99.1% pengekal kekuatan). Aliran sama ditunjukkan untuk modulus Young. Kajian ini menunjukkan asid sulfurik berpotensi untuk memperoleh gentian karbon daripada CFRP dengan 15 M memberikan keadaan paling optimum daripada segi pengekal kekuatan tegangan dan pemisahan resin epoksi.

Kata kunci: Asid sulfurik; polimer diperkuat gentian karbon dikitar semula; resin epoksi; sifat mekanikal; solvolisis

INTRODUCTION

Epoxy resins are widely used in composites due to their excellent mechanical and electrical performance. Various reinforcing agents such as carbon fibers, glass fibers and kenaf's have been incorporated to further improve

the properties and overcome their disadvantages such as brittleness (Ismail et al. 2018; Vazquez-Moreno et al. 2019; Zhao et al. 2019). Carbon fiber reinforced polymer composites (CFRP) have gained much attention due to their extensive properties such as high strength, low

density, and good resistance to corrosion (Pickering 2006). More carbon fiber composites have been manufactured and increasingly applied in industrial sectors such as aerospace, automotive, renewable energy, and sports (Rybicka et al. 2016; Su 2014). Holmes (2014) estimated that the global demand for CFRP to rise to 146 000 tonnes by 2020. Due to that, substantial amounts of composite waste generated due to manufacturing process, off-cuts as well as end-of-life products are to increase significantly in the coming years. Therefore, in order to achieve sustainable composite manufacturing, it is necessary to find ways to recycle composite waste instead of sending it to landfill or incinerator for disposal (Bai et al. 2010a). By efficient recycling, these carbon fibers could be reused in other applications by mixing it with thermoplastics to create a new composite (Mohd Ishak 2019). This will give advantages towards conserving the environment as well as reducing the cost to produce a new composite (Witik et al. 2013).

There are three common methods used to recycle composites namely, thermal, mechanical, and solvolysis processes (Oliveux et al. 2015). Other recycling methods such as microwave (Deng et al. 2019) and sonochemical (Das & Varughese 2016) treatments have also been reported. In thermal processing method, the carbon fibers were reclaimed by subjecting the composite at a high temperature to decompose polymer resin. Thermal processing includes fluidized bed reaction process (Meng et al. 2017) and pyrolysis (Jiang & Pickering 2016; Onwudili et al. 2013). This method has the ability to reclaim not only the fibers, but also the liquid and the gases decomposed from the resin which could be reused for other applications (Cunliffe et al. 2003). However, the use of high temperature (up to 700 °C) can result in fibers with lower mechanical strength retention and contamination due to char deposition (Shibata et al. 2013). Mechanical recycling involves cutting the composite into smaller pieces by means of shredding, grinding, cutting or other related mechanical processes (Kouparitsas et al. 2002; Li et al. 2016; Palmer et al. 2010). One of the disadvantages of this method is that the process results in much finer unseparated products which includes both the resin matrices and the fibers (Palmer et al. 2009).

Solvolysis has been regarded as one of the techniques that is promising in recycling composites. In a solvolysis process, solvents are used to degrade and decompose the resin to reclaim the fibers. Studies of solvolysis using supercritical solvents such as water (Bai et al. 2010b; Princaud et al. 2014) and alcohols (Jiang et al. 2009; Yan et al. 2014) have been shown promising since they could almost fully decompose all resins with the reclaimed fibers having mechanical properties comparable to the virgin fibers (Asmatulu et al. 2014). However, in order to ensure its supercritical state, this method must be carried

out in a high temperature and high-pressure environment which is very costly (Shibata et al. 2013). Other solvents have also been used for solvolysis at elevated temperatures which include sodium hydroxide (Yang et al. 2014), acetone (Keith et al. 2018) and acetic acid (Wang et al. 2015). Considering these factors, we propose a solvolysis method which uses a solvent that will reclaim the fibers at room temperature and atmospheric pressure conditions from the CFRP. To the best of our knowledge, limited studies have been reported on using solvent in solvolysis process at room temperature and atmospheric pressure to reclaim carbon fibers from epoxy composites. Previously, nitric acid was used to decompose the epoxy resin from the CFRP but it was done at a higher temperature (Dang et al. 2002). No study has reported on the use of acid at room temperature and atmospheric pressure. Therefore, in this study sulphuric acid which is a reactive acid has been proposed to be used as the solvent in the solvolysis process.

The objective of this study was to determine the lowest sulphuric acid concentration that is capable of removing all the epoxy resin while maintaining good tensile properties of the carbon fiber. Sulphuric acid at different concentrations (11 to 18 M) was used to treat the epoxy composites at room temperature and atmospheric pressure. Deionized water was used for dilution with parameters such as solvent volume (50 mL), time (30 min) and temperature (room temperature) being set constant. The reclaimed fibers from the solvolysis were then tested with single fiber tensile test to investigate the retention of their mechanical properties. Thermogravimetric analysis (TGA) was also performed on the original composite sample to determine independently its carbon fiber mass fraction, which can then be used as the reference value to compare with the mass obtained from the solvolysis process.

MATERIALS AND METHODS

In this study, the CFRP used was expired unidirectional carbon fiber reinforced epoxy resin composites (MTM58FRB/HS(24K)450-38%RW) prepreg system supplied by Solvay which was left outside the freezer and cured under room temperature for a month. Sulphuric acid (96% w/w) was used as the solvent for the solvolysis process which was supplied by QREc Chemicals. The molarity of the sulphuric acid used is 18 mol L⁻¹ (18 M). Untreated fibers were used as a comparison to the fibers obtained from solvolysis. They were extracted directly with care using tweezers from the free edge of the composite prepreg without undergoing solvolysis process.

SOLVOLYSIS OF CFRP

A small piece of CFRP of 50 × 25 × 0.5 mm was cut and put in 50 mL of concentrated sulphuric acid (18 M) which was stirred slowly using a magnetic stirrer at room

temperature. After 30 min, the sample was cleaned and washed with acetone and deionized water. The sample was then dried in an oven at 80 °C for 12 h to remove all moisture from the composite. For 17, 16, 15, 14, 13, 12, and 11 M concentrations, the sulphuric acid was diluted using distilled water according to the formula (1)

$$M_1V_1 = M_2V_2 \quad (1)$$

where M_1 is the concentration of concentrated acid (18 M); V_1 is the required volume of sulphuric acid; M_2 is the concentration of the required diluted solution (11 - 17 M); and V_2 is the volume of the diluted solution (50 mL).

The percent mass retention was calculated using (2)

$$\% \text{ mass retention} = (m_2/m_1) \times 100\% \quad (2)$$

where m_1 represent the initial mass of the composite; and m_2 is the residual mass after solvolysis.

The acid digestion process was repeated with 3 specimens for each acid concentration.

SINGLE FIBER TENSILE TEST

To evaluate the effect of solvolysis towards the carbon fiber, tensile strength of the fibers was characterized using single fiber tensile test. It was carried out using universal testing machine LLYODS instruments LR30K according to the standard ASTM-C1557 (ASTM International 2012). A single fiber filament was glued by epoxy adhesive and aligned onto a cardboard mounting tab. The mounting tab was then fixed onto the machine. The gauge length of the fiber is 20 mm and the crosshead speed were set at 1 mm min^{-1} . Both sides of the mounting tab were carefully cut at the middle-gauge point to leave the filament suspended between the grips of the machine. The carbon fiber was then loaded until failure, and the force-displacement curve was plotted. At least 25 filaments were tested for each case of acid concentration. The mounting tab and its set-up onto the tensile machine are shown in Figure 1.

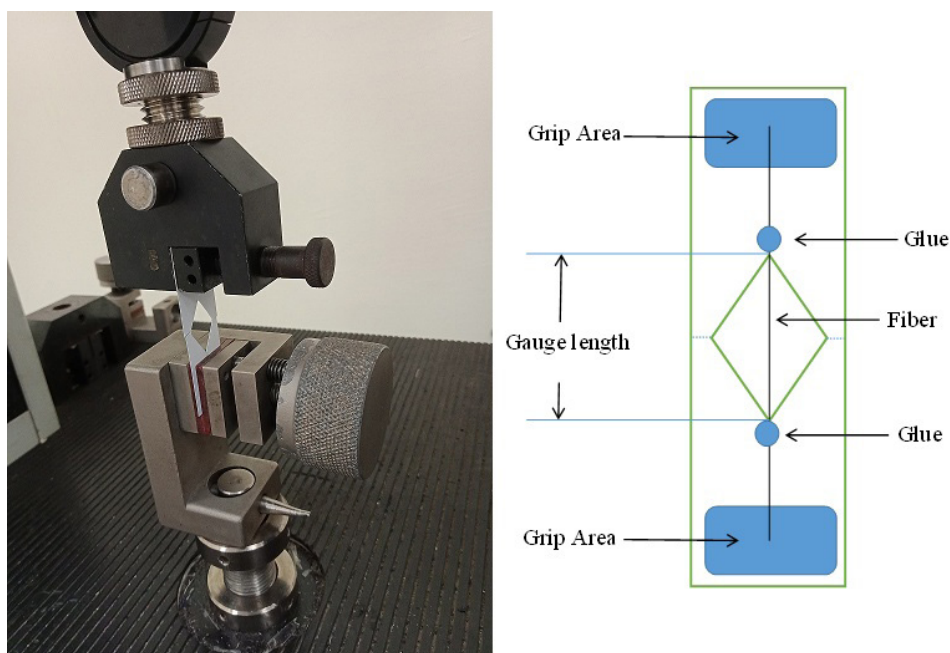


FIGURE 1. Single fiber tensile test set-up (left) and mounting tab (right)

SCANNING ELECTRON MICROSCOPY AND THERMOGRAVIMETRIC ANALYSIS

Surface characterizations and the diameter of the fibers were determined using a variable pressure scanning electron microscope (VP-SEM, JOEL iT300) while the thermal decomposition of the CFRP were performed using thermal gravimetric analyzer (Perkin Elmer, TGA 4000) from room temperature to 800 °C under nitrogen condition with a heating rate of 20 °C min^{-1} .

RESULTS AND DISCUSSION

THERMOGRAVIMETRY ANALYSIS AND PERCENTAGE OF MASS RETENTION

The TGA curve of the carbon fiber composite under nitrogen atmosphere is shown in Figure 2. TGA was used to determine independently the thermal behaviour of the epoxy resin in the composite and the reference value of the percentage of resin in the composite. From the figure,

the TGA shows a sudden drop in weight percentage at around 350 °C, indicating the rapid decomposition of the resin. It stops at around 600 °C where the decomposition process slows down and the residual weight remains constant at around 68% indicating that the resin on the composite have almost fully degraded and only carbon fibers remains. This value is consistent with the value from Table 1 for mass percent retention for the case of solvolysis at 18 M which is 69.9%, suggesting that most of the epoxy resin from the composite was completely degraded. The higher mass percent retention for the cases of acid concentrations of 17 M and below shown in

Table 1 indicates that there is increasing amount of resin residue on the fibers with decreasing acid concentration after the solvolysis process. Although there was resin residue adhering to the surface of the carbon fibers, most of the fibers could still be easily separated and extracted from the composite for further analyses, especially for the cases of acid concentration 15 M and above.

For the solvolysis using lower acid concentrations of 14, 13, 12, and 11 M, the percentage of mass retention is very high, i.e. more than 90%, which indicates that there is essentially no effective decomposition of the resin from the composites by the acid.

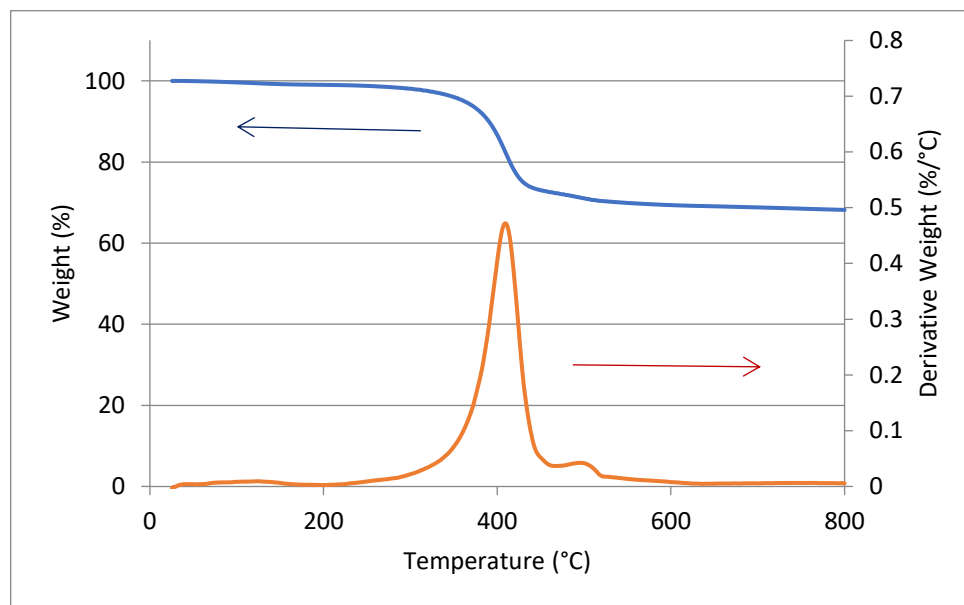


FIGURE 2. TGA curve of CFRP in nitrogen atmosphere

TABLE 1. Mass retention of CFRP from solvolysis

Sample in acid concentration (M)	18	17	16	15	14	13	12	11
Mass retention (%)	69.9	77.4	79.2	85.6	93.7	96.6	98.2	99.0

TENSILE PROPERTIES

Table 2 shows the tensile strength and the Young's modulus of the untreated fiber and fibers reclaimed from the composites through solvolysis technique at different acid concentrations. From the table, it can be observed that the tensile strength and the Young's modulus decreased

with increasing acid concentration. The highest value for the tensile strength is obtained for carbon fibers treated with acid concentration of 15 M, which is 3.43 GPa with tensile strength retention of 99.1%. As the acid concentration increased to 16 M and 17 M there is only a slight decrease of tensile strength retention of 98.6 and

96.5%, respectively. Carbon fibers in acid concentration of 18 M yield the lowest tensile strength of 3.26 GPa with tensile strength retention of 94.2%, suggesting that high concentration acid could degrade significantly the strength of the reclaimed carbon fibers.

The losses of tensile strength due to the effect of acid solvolysis are comparable in terms of percentage of tensile strength recovery with the results by other researchers who used solvolysis in recycling CFRP. Jiang et al. (2017) who pretreated the composite with nitric acid and sulphuric acid and then used potassium hydroxide as the main solvent retained 95.6% of its tensile strength while Wang et al. (2015) who used acetic acid has a tensile strength recovery of 97.6%. Yang et al. (2014)

studied the use of polyethylene glycol combined with sodium hydroxide to degrade the resin at 200 °C. They found a larger loss of tensile with strength recovery of about 93%.

As for the Young's modulus, the highest value is for carbon fibers in acid concentration of 15 M which is 226.7 GPa (97.4% modulus retention) while the lowest value is for 18 M which is 221.1 GPa (95.0% modulus retention). The values of the Young's modulus of the reclaimed carbon fibers are only slightly lower than that of the untreated fiber which is 237.7 GPa, suggesting that there is no significant difference in stiffness of the fibers after solvolysis at different acid concentrations.

TABLE 2. Single fiber tensile test results

Samples in acid concentration (M)	Diameter (μm)	Maximum load (N)	Tensile strength (GPa)	Young's modulus (GPa)
18	7.16 ± 0.12	0.1313 ± 0.0142	3.26 ± 0.35	221.1 ± 19.9
17	7.17 ± 0.15	0.1350 ± 0.0093	3.34 ± 0.23	223.8 ± 15.8
16	7.15 ± 0.22	0.1367 ± 0.0117	3.41 ± 0.29	225.1 ± 21.5
15	7.13 ± 0.20	0.1370 ± 0.0092	3.43 ± 0.23	226.7 ± 20.7
14	a	a	a	a
13	a	a	a	a
12	a	a	a	a
11	a	a	a	a
Untreated fiber	7.18 ± 0.26	0.1402 ± 0.0219	3.46 ± 0.54	232.7 ± 15.0

a: no clean fiber was successfully reclaimed

SCANNING ELECTRON MICROSCOPY (SEM) ANALYSIS

SEM is used to investigate the surface of the reclaimed fibers conditioned under various acid concentrations. It also allows the diameter of the carbon fibers to be measured directly for strength calculation. Figure 3 shows the SEM images of the reclaimed carbon fibers under the magnification of 3000 \times . No residue was observed on the surface of the carbon fibers for solvolysis using concentrated acid of 18 M. This confirms that a clean

fiber has been successfully reclaimed where all the epoxy resin was fully decomposed during the process. On the other hand, residues of epoxy resin can be clearly observed on the surface of the carbon fiber in 15 M and 16 M acid concentrations while only a small amount of resin is present in 17 M. In contrast, the micrograph of the untreated fibers, shows rough regions with significant resin adhering to it. These SEM observations demonstrate the effectiveness of the acid concentrations towards the decomposition of the epoxy resin.

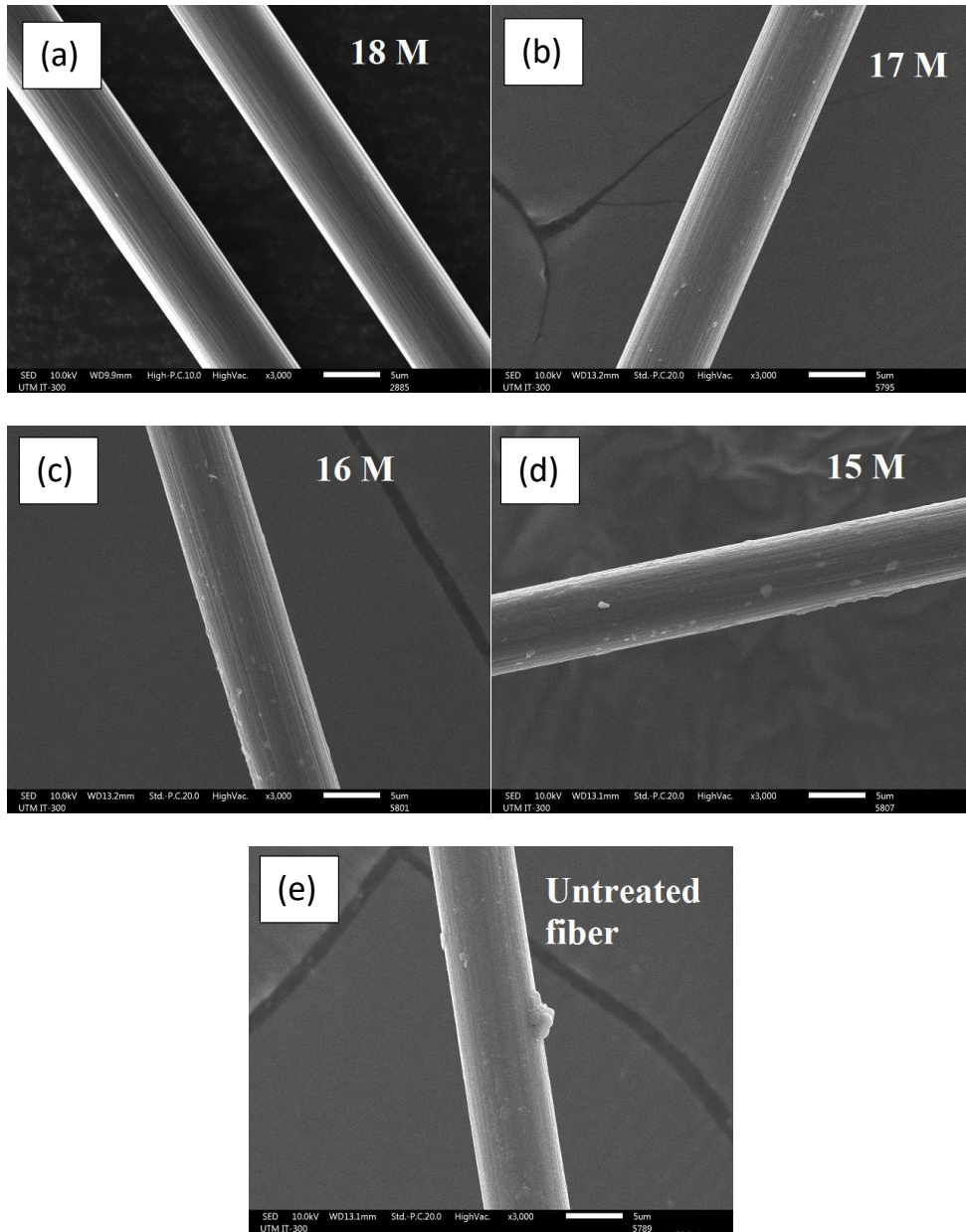


FIGURE 3. SEM micrographs of reclaimed carbon fibers using solvolysis at various acid concentrations: (a) 18 M, (b) 17 M, (c) 16 M, (d) 15 M, and (e) untreated fiber extracted from the composite prepreg

Figure 4 shows the photographs of composites after the solvolysis process at much lower concentrations of 11, 12, 13, and 14 M. For these cases, the resin is still intact and holds the carbon fibers firmly without any sign of partial

disintegration. This indicates that acid concentrations lower than 15 M are not effective for decomposing the resin from the composites under room temperature and atmospheric pressure. No further analyses were done on these samples.

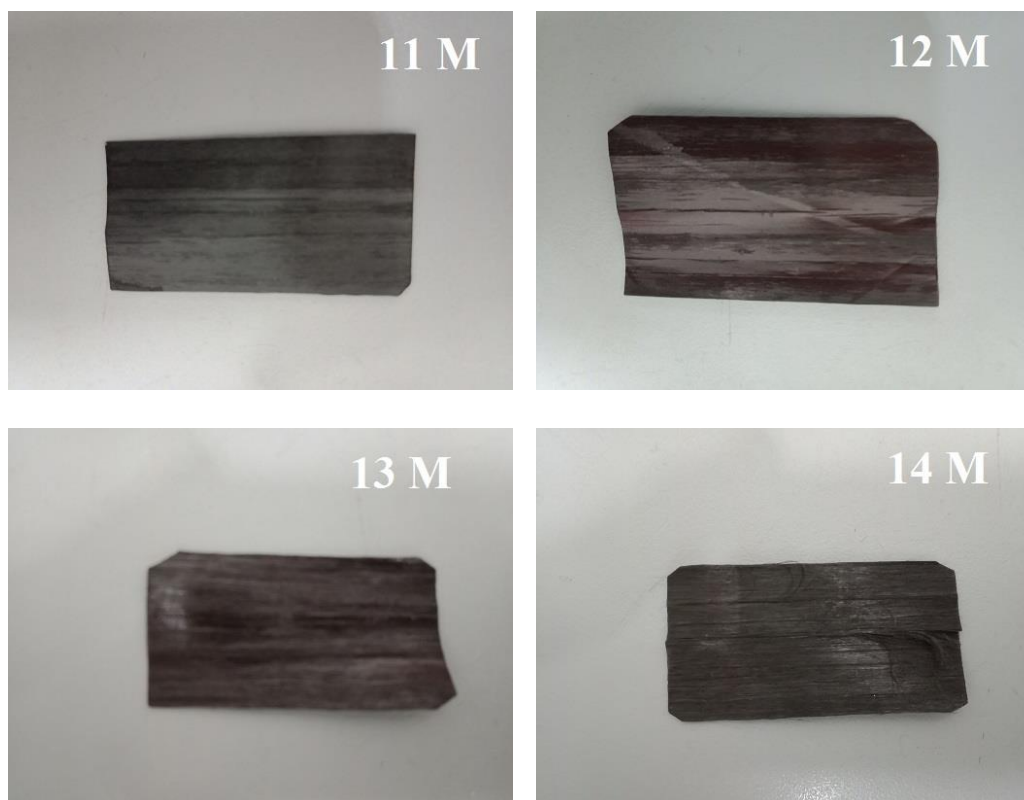


FIGURE 4. Photographs of solvolysis of carbon fiber composites using lower concentrations of sulphuric acid: (a) 11 M, (b) 12 M, (c) 13 M, and (d) 14 M

CONCLUSION

The main objective of this work was to determine the lowest sulphuric acid concentration which can remove the epoxy resin from the carbon fiber reinforced composites, at room temperature and atmospheric pressure. Sulphuric acid of concentration between 18 and 11 M was used in the solvolysis study of CFRP. It was shown that the removal of epoxy resin from CFRP increases with increasing concentration. At 18 M, almost all of the epoxy resin has been removed, giving a similar result to that obtained from TGA analysis which is 69.9% mass retention. However, at 14 M and below, epoxy resin removal was not successful at the given conditions. The single fiber tensile test of the reclaimed fibers also showed the tensile strength and Young's modulus increase with decreasing sulphuric acid concentration. At 15 M, the tensile strength and Young's modulus retention were 99.1 and 97.4%, respectively, compared to the untreated carbon fiber. No epoxy residue was observed on the surface of the carbon fibers in 18 M acid, but the amount of epoxy residue slightly increased

at lower concentrations. From the results, 15 M sulphuric acid concentration gives the optimal condition to obtain reclaimed carbon fibers based on tensile properties retention and epoxy resin decomposition. The study has shown the feasibility of solvolysis using concentrated sulphuric acid to reclaim the carbon fibers from the epoxy composites under room conditions and future studies will be looked into using concentration of sulphuric acid lower than 15 M for a longer period of time.

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