Mechanical Properties of Dental Composite Reinforced with Natural Fiber

Goh Wan Theng[1], Yanti Johari[1], Ismail Ab Rahman[1], Nor Aidaniza Abdul Muttlib[1], Mohd Nazrulhuzaimi Md Yusuf[1], Rabihah Alawi[1]*

1. School of Dental Sciences, Universiti Sains Malaysia, 16150, Kota Bharu, Kubang Kerian, Kelantan, Malaysia.

Abstract

Flexural (FS) and compressive strength (CS) of composite incorporated with kenaf fibers at ratio of 1wt% and 2wt% were tested. Four groups of samples were prepared (n=10) using SDR SureFil composite (DENTSPLY, USA) with and without incorporation of kenaf fibers, and Ever-X posterior composites (GC Corporation, Tokyo, Japan). Instron Universal Testing Machine (Shimadzu, Japan) was used. One sample from each kenaf fiber incorporated composite groups was subjected to Scanning Electron Micrograph (SEM) (FEI Quanta FEG 450, USA).

According to one-way ANOVA test, there was a statistically significant difference among four groups tested (P=0.000) for both FS and CS. Bonferroni post-hoc test showed Ever-X Posterior composites group has significantly higher FS and CS than the other groups. Both groups of composite incorporated with kenaf fibers demonstrated presence of gaps and voids between the composite and fibers. Incorporation of kenaf fibers did not increase FS and CS of the composite resin, possibly due to insufficient surface treatment of fibers to achieve bonding between fibers and composite, hence further work is required to improve the bonding.

Experimental article (J Int Dent Med Res 2019; 12(4): 1242-1247)

Keywords: Kenaf fibers, Fiber reinforced composite, Flexural strength, Compressive strength.

Received date: 03 March 2019 **Accept date: 09 June 2019**

Introduction

Dental composite resin is a widely used dental restorative material. However, when it is compared with other common restorative materials like dental amalgam, it is proven that it has less strength and fracture toughness. Therefore, there are several ways taken to increase the strength and other mechanical properties. One of the ways is the incorporation of the fibers into the dental composite. By incorporating fibers into dental composite, there was a significant increase in flexural strength and fracture toughness.[1] The fibers improve mechanical and physical properties by transferring the stress from the matrix to the fibers and acting as a crack stopper. [2]

One example of the commercially available fiber-reinforced composites is Ever-X posterior (GC Corporation, Asia) composite resin.

Corresponding author:
Rabihah Alawi
School of Dental Sciences, Universiti Sains Malaysia, Kelantan, Malaysia.
E-mail: rabihah@usm.my

The resin matrix consists of ethoxylatedbisphenol A dimethacrylate (Bis-GMA), polymethyl methacrylate (PMMA) and triethyleneglycol dimethacrylate (TEGDMA). The filler particle is 74.2 wt%, 53.6 vol% of short E-glass fiber filler and barium glass.

There are some disadvantages of synthetic fibers compared to natural fibers. A study about fiber reinforced composite stated that natural fibers are in most cases cheaper than glass fibres.[3] Natural fibers are also expected to cause less health problems for the people producing the composites compared to glass fiber based composites. This is because the natural fibers do not cause skin irritations and they are not suspected of causing lung cancer. This is a main issue since the discussion on whether very small glass fibers can cause lung cancer is yet to be concluded.[3]

The natural plants which are commonly used in applications are bast fibers, such as hemp, coir, flax, kenaf, and sisal. Kenaf is the more preferred material when compared with other natural fiber crops since it has a short plantation cycle, flexibility to environmental conditions and requires relatively lower quantity of pesticides and herbicides.[4] Akil *et al* [5] also mentioned that kenaf fiber-reinforced composites has the highest flexural strength compared to other fiber-reinforced composites (hemp, sisal and coir).

Kenaf fibers are one of the natural fibers which currently used in forming fiber reinforced composite for automotive, construction, sports and other mass production industries as they are cheaper and have higher specific strength compared to glass fibers reinforcing material.[5,6] Kenaf fibers can be easily obtained from herbaceous plant Hibiscus Cannabicus which can be found in Malaysia. The strength and stiffness of the fibers are provided by the cellulose component via hydrogen bonds and other linkages.[5] Besides, a study by Okeke *et al*, 2018 showed that inclusion of natural fibers from *Hibiscus Sabdariffa* in denture base material can increase the strength properties. [7]

However, it had been highlighted previously by Akil *et al* [5] that the lack of good interfacial adhesion between the two components (kenaf fibres and polymer matrix) and water absorption are the main concerns in the usage of kenaf fiber as a reinforcing material in composite. A study by Ochi [8] reported the presence of void and poor contact between resin and the kenaf fibers when there was insufficient resin between the fibers. According to Edeerozey *et al,*[9] poor adhesions between the fibers and the matrix, as well as the inherent high moisture absorption in the matrix could result in dimensional changes of the fibers. These may lead to micro cracking of the composite and degradation of mechanical properties. However, it has been established previously that the problems can be overcome by chemical treating the kenaf fibers using 0.6M sodium hydroxide (NaOH) without causing any significant change on the mechanical properties of kenaf fibers.[9]

Based on our knowledge, at this stage there is no fiber- reinforced dental composite resin derived from natural fibers available in the market. Therefore, in this study we will assess the mechanical properties of dental composite embedded with kenaf fibers as it has been demonstrated by the previous studies that mechanical and physical properties of the composite resin will be improved by the incorporation of fibers into the material.

Materials and methods

The materials used in this study and their compositions were listed in Table 1.

No.	Materials	Composition		Manufacturer
		Resin matrix	Inorganic filler	
1	SDR SureFil composite	TEGDMA, EBADMA	68 wt%, 44 vol%, Barium borosilicate glass	DENTSPLY,USA
2	Ever-X posterior composite	Bis-GMA, PMMA, TEGDMA	74.2 wt%, 53.6 vol% Short-E glass fibre filler, barium glass	GC CORPORATION,ASIA
3	Kenaf fibres	-	- -	

Table 1. Materials used in this study.

Experimental and control groups

The fiber reinforced composite using kenaf was tested in this study. Other commercially available fiber reinforced composite (Ever-X posterior, GC Corporation, Asia) was tested as comparison. The control sample used was the composite (SDR SureFil, Dentsply, USA). The experimental and control groups were shown in Table 2.

Preparation of composite resin incorporated with kenaf fibers.

The experimental fiber reinforced composite was prepared based on paper by Fonseca *et al* [10] with some modifications.

No.	Dental Composite	Group (n=10)		Kenaf fibre, (wt%)	CompositeResin (wt%)
1	Kenaf fibre-reinforced composite	*1A*	*1B*	*99*	*1*
		2A	*2B*	*98*	*2*
2	Ever-X posterior composite	*3A*	*3B*	*0*	*100*
3	SDR SureFil composite(control)	*4A*	*4B*	*0*	*100*

Table 2. Experimental and control groups.

Kenaf fiber modification

Kenaf fibers were treated with a surface treatment using 95% ethanol solution (GmbH) as recommended by Islam and Miao [11] and Conrad.[12] Fibers were stored at room temperature for 24 hours and left to be dried. Then, it was cut into 3mm fragments manually. All components were weighted on an analytical balance (GR-200, A&D Company Limited, Japan). After that, they were manually incorporated into the resin, according to the experimental groups as stated in Table 2.

Flexural Strength

Ten specimens of each material were prepared in 25 × 2 × 2 mm dimension using stainless steel mold. The mold was filled with composite resins (experimental fiber-reinforced composites and control restorative material), then a mylar strip and glass lid were placed over the mold and light cured for 40 seconds using LED LCU (Elipar Freelight 2, 3M ESPE, Germany) at both sides of the specimens. After polymerization, specimens were stored in distilled water at 37ºC for 24 hours. The flexural strength tests were performed using Instron Universal Testing Machine (Shimadzu, Japan) at a cross-head speed of 1 mm min-1, with 0.5 kN loading force. The specimens' thickness and height were measured using a digital caliper (Mitutoyo, Japan) before testing.

Compressive strength

Ten cylindrical specimens of each material were prepared using stainless steel mold of internal dimensions of 4 mm diameter by 6 mm height. The molds for experimental composite and control restorative material were filled and cured for 40 seconds using LED LCU (EliparFreelight 2, 3M ESPE, Germany) with a glass lid over the molds. After polymerization, each specimen was stored in distilled water at 37ºC for 24 hours. The compressive strength tests were carried out using an Instron Universal Testing Machine (Shimadzu, Japan) at a cross-head speed of 1 mm min-1, with 0.5 kN loading force. The specimens' thickness and height were measured using a digital caliper (Mitutoyo, Japan) before testing.

Scanning electron microscopy (SEM) analysis

SEM analysis was performed on the fractured 1wt% and 2wt% kenaf fiber group samples from flexural strength test (selected randomly). The samples were fixed on metal stubs, and sputtered with gold (1 cycle of 120 s) under vacuum, in a sputtering device. The surfaces were analyzed for fracture features, quantity of fiber, integrity and homogeneity along the surfaces between fibers and resin matrix using Scanning Electron Microscope (FEI Quanta FEG 450, USA).

Statistical analysis

Statistical analysis was performed using the software package SPSS version 24.0 and the results were analyzed using analysis of variance (ANOVA) and Bonferroni post-hoc tests. P value of less than 0.05 was considered as statistically significant.

Results

According to the One-way Anova test, there were significant differences in the means of flexural and compressive strength across four groups of samples (Tables 3 and 4 respectively). The samples in the group of Ever-X Posterior composites has the highest mean flexural strength followed by SDR SureFil composites, 2%wt kenaf fiber incorporated composites and 1%wt kenaf fiber incorporated composites(Table 3), whereas for compressive strength, the samples in the group of Ever-X Posterior composites has the highest mean compressive strength followed by SDR SureFil composites, 1%wt kenaf fiber incorporated composites and 2%wt kenaf fiber incorporated composites (Table 4).

Scanning Electron Microscopy (SEM) analysis

SEM analysis of 1%wt and 2%wt kenaf fiber composite were as shown in Figures 2(a)-(d).

Group	Flexural strength mean (SD)	F statistics (df)	P value
1A	112.42(23.357)		
2A	112.37(11.897)	21.674(3.000)	0.000
3A	72.40(12.505)		
4A	72.93(11.307)		

* One way ANOVA
Post hoc analysis: 1A vs 2Aand 3A vs 4A- p=1.000. Other pair comparisons- p value=0.000.

Table 3. Comparisons of flexural strength means between all the groups.

Group	Flexural strength mean (SD)	F statistics (df)	P value
1A	340.58(83.251)		
2B	210.70(21.475	43.645(3.000)	0.000
3B	147.40(10.090)		
4B	139.60(20.647)		

*One way ANOVA
Post hoc analysis: 1B vs 2B - p=1.000, 1A vs 3B- p=0.000, 1B vs 4B- p= 0.18, 2B vs 4B-p=0.06.

Table 4. Comparisons of compressive strength means between all the groups.

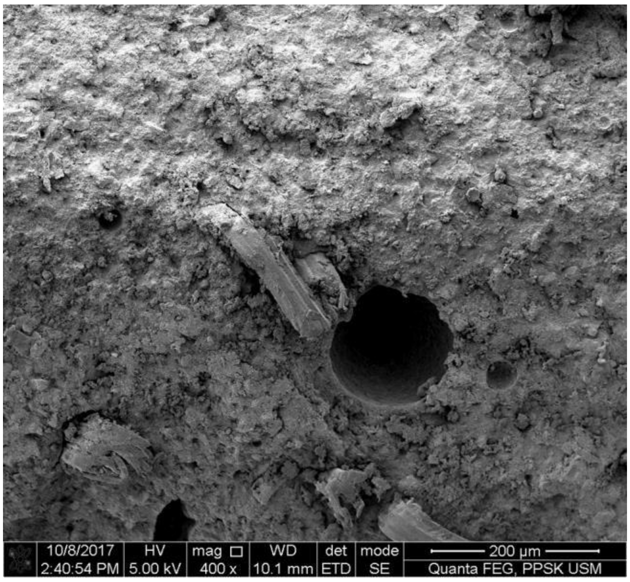

Figure 2a. SEM analysis under x400 magnification of 1%wt kenaf fiber composite sample shows there were circular empty hole which probably caused by void.

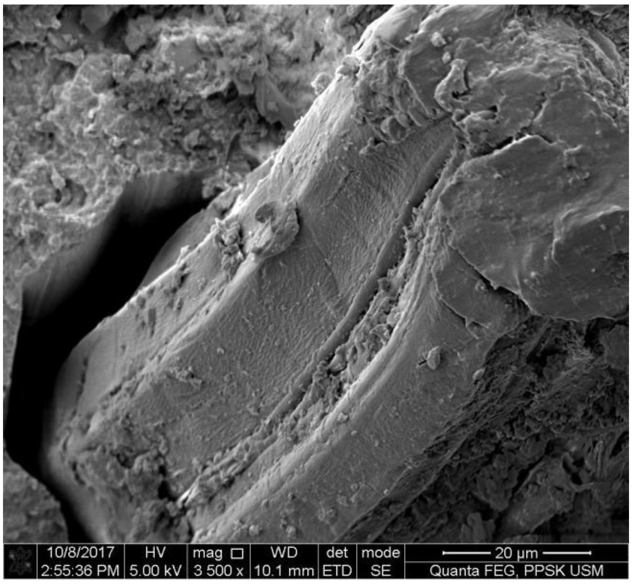

Figure 2b. SEM analysis under x3500 magnification of 1%wt kenaf fiber composite sample shows the impurities on the surface wall of the kenaf fiber.

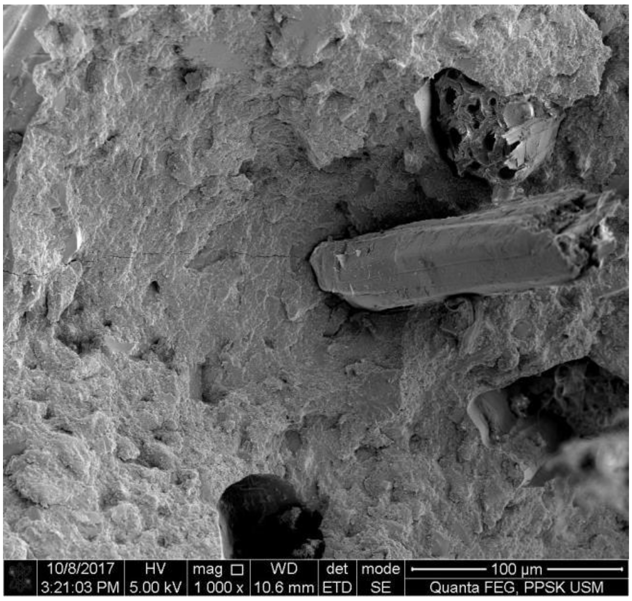

Figure 2c. SEM analysis under x1000 magnification of 2% wt kenaf fiber composite sample shows the crack propagation lines of the composite which ends at the fiber wall.

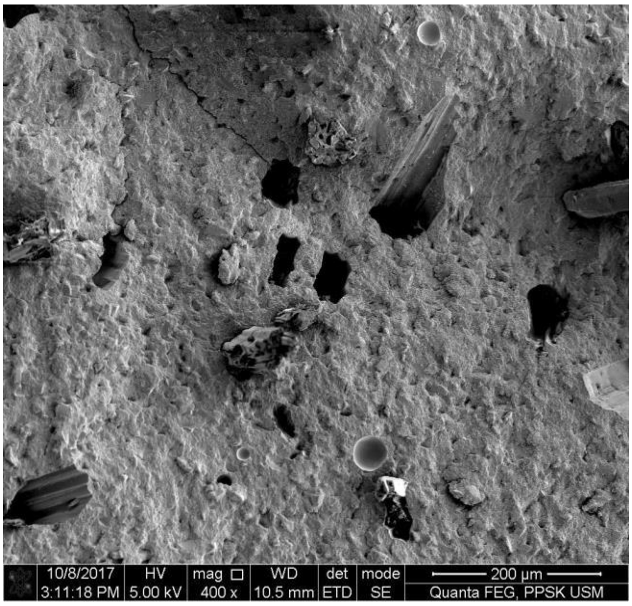

Figure 2d. SEM analysis under x400 magnification of 2%wt kenaf fiber composite sample fractured surface shows the crack propagation of the composite which ends at the holes created by fiber pulled-out.

Discussion

Many studies have demonstrated the incorporation of inorganic fiber to reinforce the mechanical properties of dental polymers.[3,13-14]

However, the outcome depends on many factors namely the quantity of fibers in the polymer matrix, the union between the fiber and resin matrix, the composition and the size of the fiber. This study evaluated the effect of incorporation of organic fiber namely kenaf fiber to dental composite resin by flexural and compressive strength tests. It was suggested that increasing fiber quantity would increase strength values.[15,16] However, the results of this study showed that the flexural and compressive strength were lower than the commercialized products.

According to the previous study of Fonseca *et al,* [10] the optimum percentage for E-glass fiber content in the composite resin is around 30% fiber by weight which achieves the best tensile strength and flexural strength results. However, kenaf fiber (0.75g/m3) [17] is extremely less dense compared to the E-glass fiber (2.55 g/cm3) (AZO Materials). Thus, the desired percentage of fiber content in the composite resin described in study of Fonseca *et al* [10] was impossible to achieve looking at the high volume of the kenaf fiber to be incorporated into small volume of composite resin by mixing manually using plastic spatula. The ratio of 1% kenaf fiber to 99% of composite (SureFil SDR) was then used as the starting ratio of the sample group, followed by 2% kenaf fiber to 98% composite (SureFil SDR) resin ratio. It was proved that increase in the fiber percentage might improve the mechanical properties of kenaf fiber in the study by Vilay *et al.* [16]

According to a study by Pickering *et al,*[18] fiber length plays a crucial role in fiber-reinforcement of composite, especially affecting the mechanical properties of the material. This is due to the transferring of the tensile load from the matrix into the fiber at the interface of fiber and matrix. The tensile stress of the fiber at the ends is zero but increase along the fiber length. Therefore, the increase in fiber length commonly increases fiber load bearing efficiency. However, overlay long fiber may cause tangling during mixing. This will cause poor fiber dispersion which then causes lower effect of overall composite-reinforcement. The highest upper bound value as mentioned in the study is around 3mm in length. Therefore, the fiber length we used in this study was 3mm.

In this study, the kenaf fiber orientation in the composite resin was random and discontinuous. Commonly, random discontinuous orientation of fiber is used in the sites where multidirectional force is applied. The type of orientation and length of the fiber in composite manufacturing depend on the level and nature of the applied stress as well as the cost of production. However, aligned fibrous composites are inherently anisotropic, in that the maximum strength and reinforcement are achieved along the alignment (longitudinal) direction. This is because the aligned layers that are fastened together one on top of another at different orientations are frequently used when multidirectional stresses are imposed within a single plane. [19] There was a study showed that kenaf fiber in woven structure gives high tensile strength and impact resistance properties.[20] Hence, further study on the effect of change in kenaf fiber orientation on the mechanical properties shall be done to enhance it in kenaf fiber reinforced composite resin.

The result of the SEM analysis in this study showed the crack propagation on composite resin stops at the ruptured fiber and the obvious gap between the fiber and the composite resin. This suggested that the surface adhesion of the kenaf fiber was insufficient. In the study by Edeerozey *et al* [7] and Mishra *et al,* [21] the surface impurities and lack of surface adhesion of kenaf fiber was one of the causes of low tensile properties of the kenaf fiber reinforced composite.

In this study, the surface treatment of kenaf fiber was done using with 95% ethanol solution (GmbH) according to Islam and Miao [11] and Conrad [12] to remove the waxy substance on the fiber surface. Although it is possible to have strong interfacial bonding, plant-based fiber composites generally have limited interaction. This is due to the hydrophilic properties of the fiber and hydrophobic properties of the matrices. As it is commonly known that dental composite resin is moisture sensitive, slight moisture is enough to reduce the bonding of the material. Thus, wettability can be considered as an essential precursor to bonding.[18] Further attempt of chemical modifying the kenaf fiber according to Edeerozey *et al* [9] or heat treating the kenaf fiber according to Ariawan *et al*[22] can be done for better result.

In addition, as mentioned in the result of the Scanning Electron Microscopy, porosity was one of the findings. Porosity in the composite is one of the important factors to influence the mechanical properties of the material. The porosity mainly caused by the inclusion of air during processing, limited wettability of fibers, difficulty in compacting fiber, lumens as well as hollow features in fiber.[23] As the geometrical compaction limit is exceeded, the greater the fiber content, the greater the porosity.

Conclusions

Incorporation of 1% and 2% of kenaf fibers did not increase flexural and compressive strength of the composite resin, possibly due to insufficient surface treatment of fibers to achieve bonding between fibers and composite. Hence further work is required to improve the bonding.

Declaration of Interest

The authors declare no conflicts of interests. The study was supported by 'Short term grant, No. 304/PPSG/61313110' of Universiti Sains Malaysia and School of Dental Sciences, USM KPI's fund. The authors would like to thank Mr. Mohd Yusuf Soon Abdullah, the medical laboratory technologist of Biomaterial Lab in School of Dental Sciences Universiti Sains Malaysia for the technical support. We also would like to thank all laboratory staffs for guiding us through the entire process, and Assoc. Prof Dr Wan Muhamad Amir Wan Ahmad for statistical consultation

References

1. Xu X, Xu HHK. Dental Composite Reinforced with Ceramic Whiskers and Nanofibers. In: Handbook of Nanomaterials Properties. Berlin: Springer Berlin Heidelberg; 2014: 1299-1320.
2. Garoushi S, Säilynoja E, Vallittu PK, Lassila L. Physical properties and depth of cure of a new short fibre reinforced composite. Dent Mater 2013;29(8):835-41.
3. Jawaid M, Abdul Khalil HPS. Cellulosic/synthetic Fiber Reinforced Polymer Hybrid Composites. Carbohyd Polym 2011; 86: 1-18.
4. Nishino T, Hirao K, Kotera M, Nakamae K, Inagaki H. Kenaf Reinforced Biodegradable Composite. Compos Sci and Technol 2003; 63(1):1281–86.
5. Akil HM, Omar MF, Mazuki AAM et al. Kenaf fibre Reinforced Composite: A Review. Mater Design 2011; 32(1):4107–21.
6. Anuar H, Zuraida A. Improvement in Mechanical Properties of Reinforced Thermoplastic Elastomer Composite with Kenaf Bast Fiber. Composites Part B 2011; 42(1):462-5.
7. Okeke KN, Vahed A, Singh S. Improving the Strength Properties of Denture Base Acrylic Resins Using Hibiscus Sabdariffa Natural Fiber. J Int Dent Med Res 2018;11(1):249-54.
8. Ochi S. Mechanical Properties of Kenaf fibres and Kenaf/PLA Composites. Mater Design 2007; 40:446-52.
9. Edeerozey A, Akil HM, Azhar A, Ariffin M. Chemical Modification of Kenaf Fibers. Mater Lett 2007;61:2023–5.
10. Fonseca RB, de Almeida LN, Mendes GAM, Kasuya AVB, Favara˜o IN, de Paula MS. Effect of Short Glass Fibre/filler Particle Proportion on Flexural and Diametral Tensile Strength of A Novel Fibre-Reinforced Composite. J Prosthodont Res 2016;60(1):47-53.
11. Islam MS, Miao M. Effect of Removing Polypropylene Fiber Surface Finishes on Mechanical Performance of Kenaf-polypropylene Composites. Composites Part A 2011;42(11):1687-93.
12. Conrad CM. Determination of Wax in Cotton Fiber - A New Alcohol Extraction Method. Ind Eng Chem Anal Ed 1944;16 (12):745–8.
13. Pan Y, Liu F, Xu D, Jiang X, Yu H, Zhu M. Novel Acrylic Resin Denture Base with Enhanced Mechanical Properties by The Incorporation of PMMA-modified Hydroxyapatite. Prog Nat Sci – Mater 2013; 23(1):89-93.
14. Herrera-Franco PJ, Valadez-Gonza´lez A. A Study of The Mechanical Properties of Short Natural-fiber Reinforced Composites. Composites Part B. 2005; 36:597-608.
15. Sukumar A, John E. Fiber Addition and Its Effect on Concrete Strength. Int J Innov Res Adv Eng 2014;1(8):145-9.
16. Vilay V, Mariatti M, Mat Taib R, Todo M. Effect of Fiber Surface Treatment and Fiber Loading On The Properties of Bagasse Fiber–reinforced Unsaturated Polyester Composites. Compos Sci Technol 2008;68:631–8.
17. Ribot, N.M.H., Ahmad, Z., Mustaffa, N.K. Mechanical Propertise of Kenaf Fibre Composite Using Co-cured In-line Fiber Joint. Int J Eng Sci Technol 2011;3(4):3526-34.
18. Pickering KL, Efendy MGA, Le TM. A Review of Recent Developments in Natural Fiber Composites and Their Mechanical Performance. Composites Part A 2016;83:98-112.
19. Callister W. D. Composites, Material Science and Engineering: An Introduction. 7th ed. USA: John Wiley & Sons, Inc; 2006: 585-86.
20. Yahaya R, Sapuan SM, Jawaid M, Leman Z, Zainudin ES. Effect of Fiber Orientations on The Mechanical Properties of Kenaf-Aramid Hybrid Composites for Spal-liner Application. Defence Technol 2016; 12(1):52-8.
21. Mishra S, Mohanty AK, Drzal LT et al. Studies on Mechanical Performance of Biofibre/Glass Reinforced Polyester Hybrid Composites. Compos Sci Technol 2003; 63:1377-85.
22. Ariawan D, Mohd.Ishak ZA, Mat Taib R, Ahmad Thirmizir MZ, Phua YJ. Effect of Heat Treatment on Properties of Kenaf Fiber Mat/Unsaturated Polyester Composite Produced by Resin Transfer Molding. Appl MechMater 2015:699:118
23. Madsen B, Thygesen A, Lilholt H. Plant Fiber Composites-Porosity and Stiffness. Compos Sci Technol 2009; 69(7-8):1057-69.