

**INTERFACIAL CHARACTERISATION OF NATURAL FIQUE
FIBRE/POLYPROPYLENE COMPOSITES USING SINGLE FIBRE
FRAGMENTATION TEST**

Ross F. Minty^{a*}, Diego Martinez de Luca^b and Meisam Jalalvand^b

^aDepartment of Mechanical and Aerospace Engineering, University of Strathclyde, Glasgow,
G1 1XJ, United Kingdom

^bFaculty of Engineering and Physical Sciences, University of Southampton, Southampton,
SO17 1BJ, United Kingdom

*ross.f.minty@strath.ac.uk

ABSTRACT

The present work focuses on a comprehensive investigation into the fibre-matrix interface of fique fibre-reinforced polypropylene (PP) composites using the single fibre fragmentation test. The objective was to establish the degree of adhesion that exists between fique fibre and PP prior to subsequent surface treatment, which is targeted for a following study.

INTRODUCTION

There is growing demand for increasingly more sustainable fibre-reinforced composite materials for use in higher performance applications. Reinforcement of polymer matrix composites with natural fibres is increasingly being investigated due to the inherent benefits they can bring in terms of biodegradability, natural occurrence, low cost, and relative lack of impact on the environment when compared to their glass and carbon counterparts. As such the demand to produce natural fibre-reinforced composites that possess superior mechanical properties has never been higher, with a great level of investment having been placed into research with the goal of broadening our understanding of how to optimize mechanical performance.

If the performance of such materials is to be maximised, then it is critical to optimize the stress transfer capability of the interfacial region which exists between the reinforcement fibre and the polymer matrix. Historically this area has been a significant challenge when it comes to natural fibres, with limited surface adhesion typically resulting in poor wetting of the fibre and the resultant mechanical properties of the composite.

There has been significant work done in the literature focused on studying the adhesion of various natural fibres with both thermoplastic [1–9] and thermosetting resin systems [8,10–13], along with the potential implications it can have on the material properties of the composite [14–18]. However, compared to flax, hemp and jute fibres, there has been relatively little investigation into the use of fique fibres [19–23], particularly at the fibre-matrix interface level. Among the natural reinforcement fibres, fique appears to offer significant promise due to its high mechanical strength [23] ready availability in the form of nonwoven mats produced in Colombia [21]. This paper thus focuses on the design and development of a single fibre

fragmentation technique to study the fibre-matrix interface strength between untreated fique fibres and polypropylene (PP).

EXPERIMENTATION

Materials

The experiments were carried out untreated fique (*Furcraea andina*) fibre supplied by Compania de Empaques through Universidad Nacional de Colombia. The average diameter of the fibre was approximately 148 μm and the nominal average strength was 330 MPa [24]. The polypropylene used for the manufacture of the samples was purchased in thin sheet form from RS components and used as received.

Sample Preparation

The stepwise approach for manufacturing samples was largely based upon the approach used by Awal et al [9]. A specialized mould, manufactured from 8 mm thick aluminium 6082-T6 via waterjet cutting, was used to press mould the single fibre fragmentation test (SFFT) samples. Steel shims 0.5 mm thick were cut to fit within the mould to allow for placement of the fique fibres at the correct depth and allow for fibres to be secured using tape. An exploded diagram of the mould is provided in Figure 1.

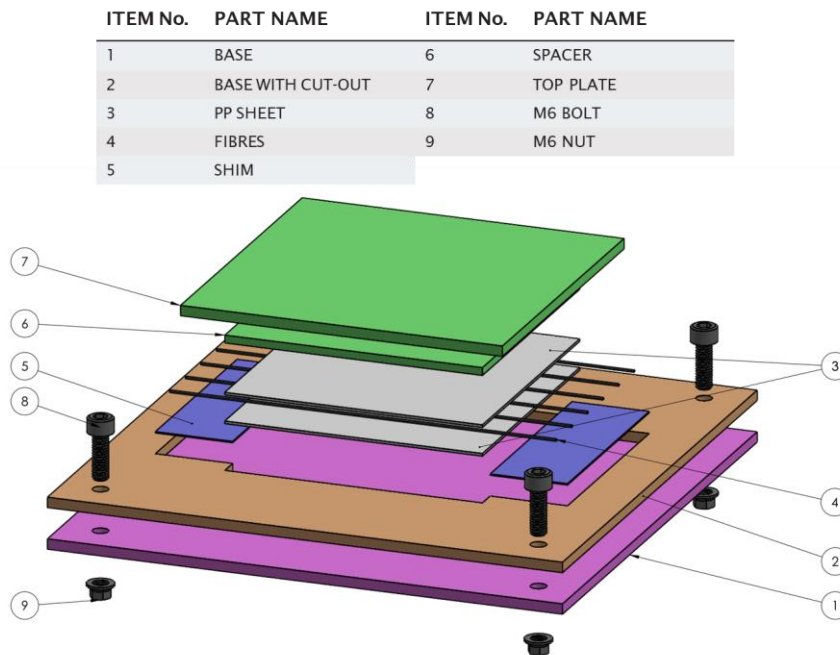


Figure 1: Exploded diagram of the mould used for manufacturing the samples

The specific manufacturing procedure is described in detail below.

- i. One PP sheet with a thickness of 0.5 mm was placed between the steel shims within the aluminium mould.

- ii. Individual single fibre fibres were then separated manually from a bundle. Proper care was given not to damage the fibres during isolation, as well as to ensure that the exposed sections of fibre to be used for the test were protected from contamination, whether through surface or human contact. Fibres were then cleaned by submerging them in warm water for 30 minutes, and subsequently dried in an oven at 80 °C for 50 minutes. Both fibre ends were secured to the steel shims using adhesive tape to keep the fibres straight along the longitudinal axis of the PP sheet and preserve fibre alignment during press processing.
- iii. A second PP sheet was then placed over the first PP sheet with the fibres. Once all the fibres were in place, it was made sure that all the fibres were parallel to each other along the length axis of the PP sheet as shown in Figure 2.
- iv. Once this was completed, the weighted top plate was placed on top of the mould (total applied pressure 4 MPa), and the mould was placed in an oven preheated to 200 °C.
- v. The mould was then left in this state for 25 minutes.
- vi. Following this the mould was removed from the oven and allowed to air cool until it reached room temperature. The top plate was then removed, and the samples released from the steel shims producing samples as shown in Figure 3.
- vii. Only straight fibres were considered for further tests. The samples were cut into the desired geometry to fit the DEBEN MT2000 test rig used for the test. Final specimen thickness was 1 mm and was measured using a micrometer. The chosen gauge length for the samples was 33 mm and the gauge width was 20 mm.

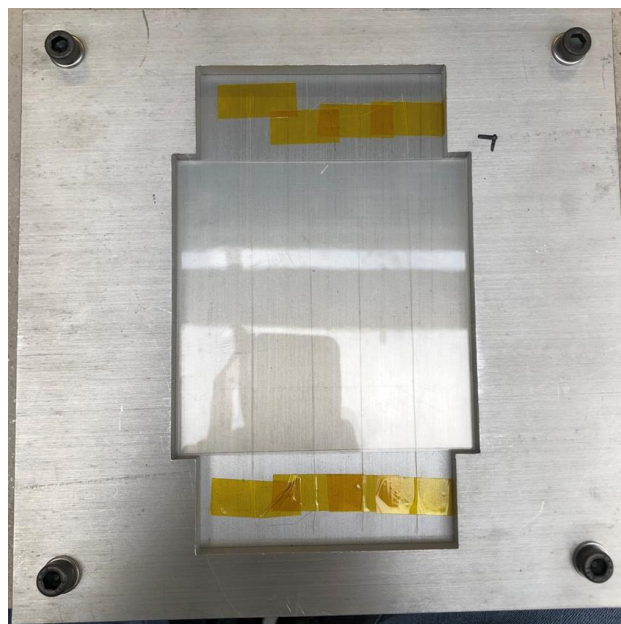


Figure 2: PP sheets and fibres placed in the mould prior to placing top plate.

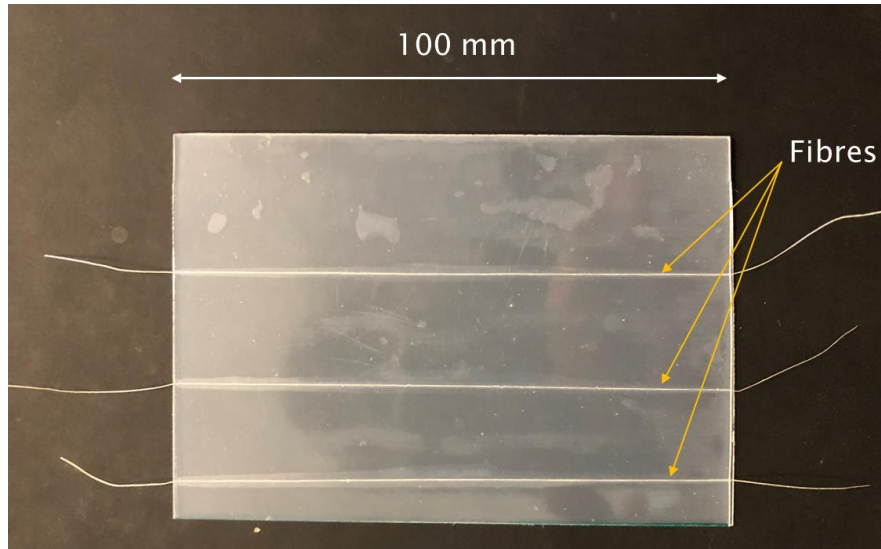


Figure 3: Single fibre samples following removal from mould.

Single Fibre Fragmentation Procedure

The single fibre fragmentation test was carried out using a DEBEN MT2000 microtensile test rig. The rig is capable of running tensile and compression tests under displacement control conditions, or constant load with cyclic tests also possible. For the SFFT tests, the tensile load was applied at a constant strain rate of 0.4 mm/min to the sample until the fibres breaking point. Loading was stopped if the specimen failed, or when the fragmentation saturation level was achieved [9].

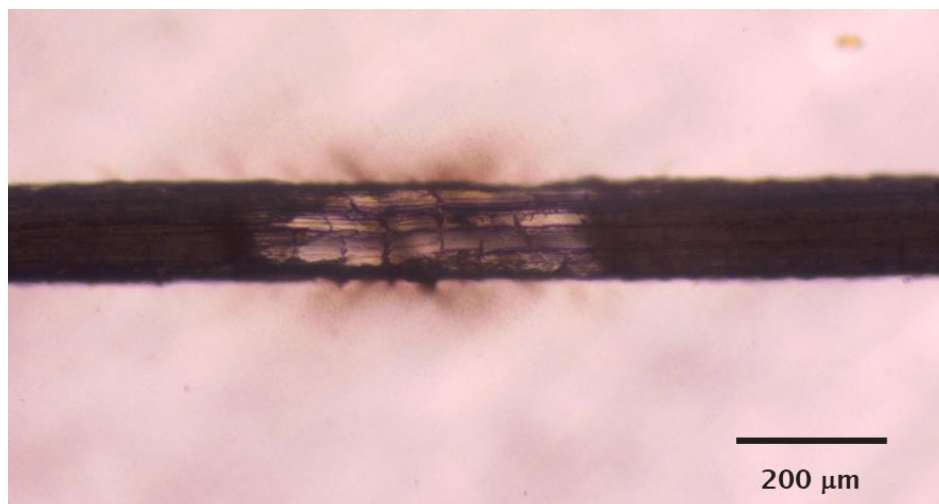


Figure 4: Fibre fragmentation shown following testing and sample polishing.

Following testing, samples were removed from the test rig and polished before then being observed under 100x, and 200x magnification using a GT Vision GXM-L3230BD, and Olympus BH-2 microscope. Each sample was analysed and photographed along the entire gauge length, with fibre fragmentations like that shown in Figure 4 noted and counted for

calculating the fragment lengths. With the number of fragments within the gauge length known, the critical fibre length could be calculated according to [25]:

$$l_c = \frac{4}{3} \times \frac{\text{Sample Gauge Length}}{\text{No. of Fragments within gauge}}$$

The values for apparent Interfacial Shear Strength (IFSS) could be calculated according to [25]:

$$IFSS (\tau) = \frac{r_f \sigma_f}{l_c}$$

Where r_f is the radius of the fibre, σ_f the average tensile strength of the fibre and l_c the critical fibre length defined previously. To calculate the approximate radius of each fibre, the samples were cross sectioned at multiple places along the gauge length and the cross-sectional area of the fibre measured. From this an approximation could be made for the average fibre radius value for the apparent IFSS calculation. For the purposes of this investigation the average tensile strength of the fibre was assumed to be 330 MPa.

RESULTS

At the time of writing, 24 samples have been tested using the procedure defined in section 2, producing the data defined in Figures 5 and 6 respectively. Figure 5 provides the critical fibre length values measured for each of the samples tested. The average critical fibre length measured was 10.83 mm, with a standard deviation of 3.06 mm.

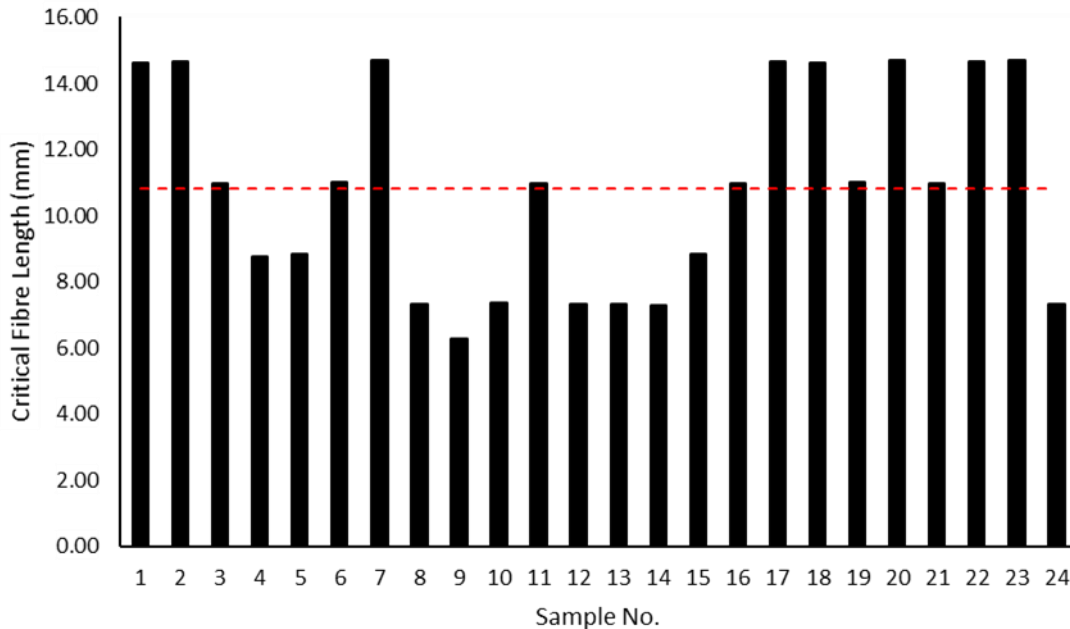


Figure 5: Critical fibre lengths values calculated for samples studied.

Figure 6 shows the values for IFSS calculated for each of the samples studied. The average IFSS value calculated was approximately 2.5 MPa, with a standard deviation of 0.7 MPa. This result is not dissimilar from the 4.5 MPa reported by Awal et al [9] for flax fibre/PP composites and values of 3 to 6 MPa for bare glass fibre/PP reported in the past [26]. However it has been observed in the past to be possible to reach IFSS values of 8 to 12 MPa [9,27] for flax/PP composites and 11 to 14 MPa for glass fibre/PP composites [28] when surface treatment or matrix modification has been applied.

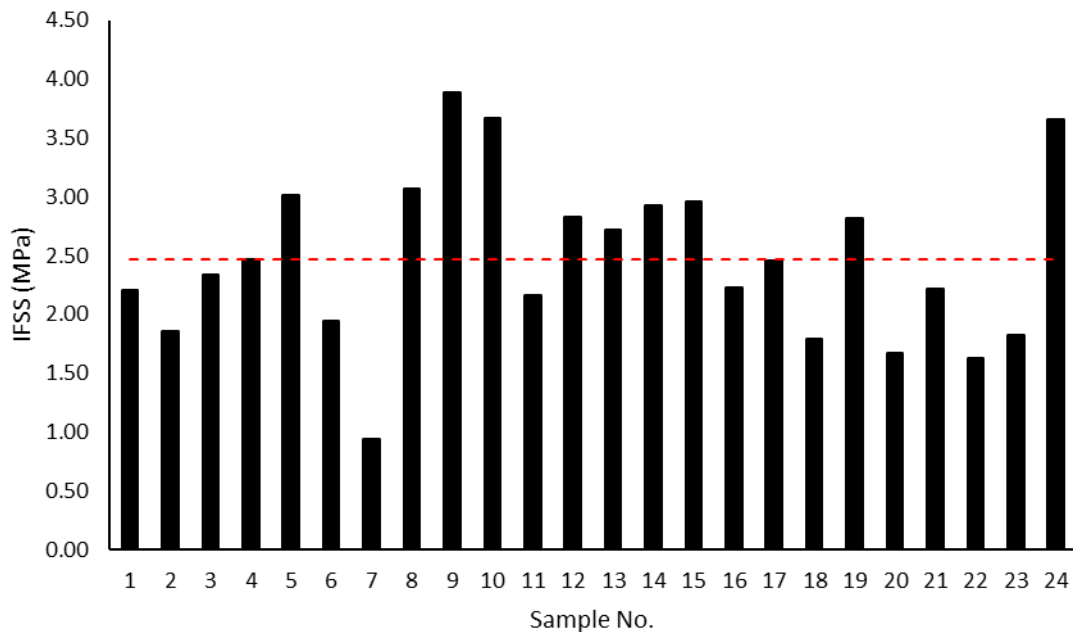


Figure 6: Apparent IFSS values measured for samples studied.

Overall, the degree of adhesion that exists between the untreated fique fibres and the PP matrix would appear to have been poor. Poor coupling is suggested by the nature of the fragmentations as shown in Figure 4, with debonding observed and little to no transverse cracking occurring [28].

CONCLUSIONS

In this study, SFFT tests have been conducted to characterise the fibre–matrix adhesion between natural, untreated fique fibres with a PP matrix. An in-house testing technique was developed to allow for critical fibre length values and interfacial strength values to be obtained and analysed. The results presented in this paper show that the degree of adhesion between untreated fique fibres and PP is comparable to other untreated natural fibres studied in the past, along with untreated bare glass fibre. However, there would still appear to be significant potential for improvement of the IFSS through surface modification of the fique fibres to improve the degree of adhesion with the PP matrix. This has shown to be the case for other comparable fibres in the past and thus would justify further study. Furthermore, it could be beneficial to conduct further research into the fibre-matrix interface of fique fibres with other

prominent matrix systems to evaluate performance compared to other reinforcement fibres. Both would fill gaps that currently exist within the current knowledge base.

ACKNOWLEDGEMENTS

This work was funded under The British Council Newton Fund Institutional Links Grant Agreement 527677533 in collaboration with ICIPC and National University of Colombia. The data necessary to support the conclusions are included in the paper.

REFERENCES

- [1] J. L Thomason, J.L. Rudeiros-Fernández. *Compos Interfaces*. 29 (2022) 175–96.
- [2] T. Huber, J. Müssig. *Compos Interfaces*. 15 (2008) 335–49.
- [3] J. Gassan. *Compos - Part A Appl Sci Manuf*. 33 (2002) 369–74.
- [4] A. le Duigou, J. Merotte, A. Bourmaud, P. Davies, K. Belhouli, C. Baley. *Compos Sci Technol*. 151 (2017) 228–33.
- [5] N. E. Zafeiropoulos. *Compos Interfaces*. 14 (2007) 807–20.
- [6] F. G. Torres, M. L. Cubillas. *Polym Test*. 24 (2005) 694–8.
- [7] S. O. Amiandamhen, M. Meincken, L. Tyhoda. *Fibers Polym*. 21 (2020) 677–89.
- [8] Y. Xie, C. Hill, Z. Xiao, H. Militz, C. Mai. *Compos Part A Appl Sci Manuf*. 41 (2010) 806–19.
- [9] A. Awal, G. Cescutti, S. B. Ghosh, J. Müssig. *Compos Part A Appl Sci Manuf*. 42 (2011) 50–6.
- [10] J. P. Craven, R. Cripps, C. Viney. *Compos Part A Appl Sci Manuf*. 31 (2000) 653–60.
- [11] S. J. Eichhorn, R. J. Young. *Compos Sci Technol*. 64 (2004) 767–72.
- [12] C. Baley, F. Busnel, Y. Grohens, O. Sire. *Compos Part A Appl Sci Manuf*. 37 (2006) 1626–37.
- [13] R. Joffe, J. A. Andersons, L. Wallström. *Compos Part A Appl Sci Manuf*. 34 (2003) 603–12.
- [14] P. J. Herrera-Franco, A. Valadez-González. *Compos Part A Appl Sci Manuf*. 35 (2004) 339–45.
- [15] F. Sarasini, J. Tirillò, C. Sergi, M. C. Seghini, L. Cozzarini, N. Graupner. *Compos Struct*. 88 (2018) 394–406.
- [16] M. Abdelmouleh, S. Boufi, M. N. Belgacem, A. Dufresne. *Compos Sci Technol*. 67 (2007) 1627–39.
- [17] K. L. Pickering, M. Efendy, T. M. Le. *Compos Part A Appl Sci Manuf*. 83 (2016) 98–112.
- [18] C. Baley, A. Bourmaud, P. Davies.. *Compos Part A Appl Sci Manuf*. 144 (2021).
- [19] M. F. Muñoz-Vélez, M. A. Hidalgo-Salazar, J. H. Mina-Hernández. *Polymers*. 10 (2018) 1–14.
- [20] J. H. M. Hernandez, E. F. T. Perea, K. C. Mejía, C. Jacobo. *Polymers*. 12 (2020) 1–15.

SAMPE Europe Conference 2022 Hamburg - Germany

- [21] M. A. Hidalgo-Salazar, J. H. Mina, P. J. Herrera-Franco. *Compos Part B Eng.* 55 (2013) 345–51.
- [22] C. Gómez Hoyos, A. Vázquez. *Compos Part B Eng.* 43 (2012) 3120–30.
- [23] S. Delvasto, E. F. Toro, F. Perdomo, R. M. de Gutiérrez. *Constr Build Mater.* 24 (2010) 187–92.
- [24] B. Casares Fernández, M. Jalalvand, J. Kelly. *CompTest 2021* (2021).
- [25] S. Feih, K. Wonsyld, D. Minzari, P. Westermann, H. Lilholt. *Forskningscenter Risø.* (Denmark. Forskningscenter Risoe. Risoe-R; No. 1483(EN)).
- [26] L. Yang, J. L. Thomason. *Compos Part A Appl Sci Manuf.* 41 (2010) 1077–83.
- [27] H. L. Bos. *Technische Universiteit Eindhoven* (2004).
- [28] P. Nygård, K. Redford, C. G. Gustafson. *Compos Interfaces.* 9 (2002) 365–88.