



Extraction and Characterization of Natural Lignocellulosic Fiber from *Borassus flabellifer* Leaf Sheath

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A natural lignocellulosic fiber has been extracted from *Borassus flabellifer* leaf sheath (BFSE) fibers and they characterized for its chemical composition, morphology, crystallinity, density, single fiber tensile strength and thermal property through standard analytical method using Fourier-transform infrared spectroscopy (FTIR), scanning electron microscope (SEM), X-ray diffraction (XRD), pycnometer, universal testing machine and thermogravimetric analysis (TGA). The chemical compositions of BFSF were found as cellulose, hemicellulose and lignin contents of 48.20 ± 0.95 , 25.92 ± 0.83 and $23.83 \pm 0.34\%$, respectively. The SEM micrograph confirmed the presence of textured surface of fiber. TGA indicates that the fibers are thermally stable up to 208 °C. The crystallinity and single fiber tensile strength were calculated as 33.15% and 170-220 MPa, respectively.

Keywords: *Borassus flabellifer* leaf sheath, Lignocellulosic fiber.

INTRODUCTION

Over the past few decades synthetic fibers have been used as reinforcements in polymer composites for several applications [1]. While manufacturing to end use, synthetic fibers trigger volatile organic compound (VOC), carbon emission and particulate emission, which are potential hazardous to environment. The increasing attention on the stringent environmental legislations and ecological economic consequences drive to minimize or nullifies the usage of synthetic fibers. The natural fibers are being considered as an alternate for synthetic fibers due to their low density, less machine wear, low cost, high specific strength, good thermal and acoustic insulator, biodegradability, renewable and sustainable resources [2,3].

The natural fibers have been classified as animal, plant and mineral fiber based on the main source [4]. Among natural fibers, the mineral fibers asbestos is hazardous to human and hence it is restricted to use [5] whereas the animal fibers such as silk and wool are more expensive than plant fibers and therefore the usage of plant fibers is mostly economical. The Palmyra palm tree *Borassus flabellifer*, a multipurpose tree of great utility of making food, beverage, sugar, fiber, construction

material and fuel, is one of the genres of Arecaceae (Palmaceae) family widely found in Asian countries [6]. They give fibers from fruits, leaf stalk or petiole and leaf sheath. A comprehensive study on morphology, mechanical and thermal properties of fruit fibers have already been reported and it is found that the mechanical properties of these fine fibers are superior to the coarse fiber [7]. The studies on the chemical composition and mechanical properties of *Borassus flabellifer* leaf stalk fibers and their utilization as the reinforcement in polymer composites has also been carried out [8,9]. However, the chemical composition, morphology, crystallinity, mechanical properties and thermal properties of *Borassus flabellifer* sheath fibers (BFSF) have not studied so far. In this study, *Borassus flabellifer* leaf sheath fibers are extracted and investigated for the use of prospective reinforcement in polymer composites.

EXPERIMENTAL

Borassus flabellifer sheath fibers (BFSF) used in this study were obtained from *Borassus flabellifer* tree located in southern district of Tamilnadu state, India (9.10°N 77.42°E). The sheaths were cut from the trunk of tree and the stalk portion was removed

by sharp knife. Then they were hammered gently and the fibers were separated from the pith by combing process. Thus, obtained fibers were washed in ethanol, dried at room temperature for a week and used in this study.

Moisture content: The weighed quantities of BFSF (M_0 , g) were placed in an electric oven at the temperature range of 120 ± 5 °C for 2 h. The weight of fiber taken from the oven (M_1 , g) were measured by an electronic balance with the precision of 0.1 mg and the percentage of moisture was calculated using the following formula:

$$\text{Moisture content (\%)} = \frac{M_1 - M_0}{M_0} \times 100 \quad (1)$$

Chemical composition: *Borassus flabellifer* sheath fibers (BFSF) were characterized for the chemical composition of macromolecules such as cellulose, hemicellulose and lignin content by standard analytical methods as reported earlier [10]. Five random samples were analyzed and the average of values was taken.

Fibre density: The fiber density was determined by liquid displacement method using pycnometer with toluene [11]. The *Borassus flabellifer* sheath fibers (BFSF) were cut into small pieces and then put into a pre-weighed pycnometer specific gravity bottle with capillary Teflon stopper. The vacant rest of the volume of bottle was filled with toluene. The fiber density was calculated using the following formula:

$$\rho_{\text{bfsf}} = \frac{(m_2 - m_1)\rho_t}{(m_3 - m_1)(m_4 - m_2)} \quad (2)$$

where, ρ_{bfsf} = density of BFSF (g/cm^3); ρ_t = the density of toluene (g/cm^3); m_1 = the mass of empty pycnometer (g); m^2 = the mass of pycnometer with fibers (g), m^3 = mass of pycnometer with toluene (g); and m_4 = mass of pycnometer with fibers and toluene (g).

FTIR analysis: Perkin-Elmer spectrum RXI Fourier Transformer Infra-Red spectrometer was used to derive the FTIR of *Borassus flabellifer* sheath fibers (BFSF) in KBr matrix in the wavenumber range of 4000-600 cm^{-1} . The chopped samples were grounded with KBr to obtain fine powder using a mortar and pestle. They were pelletized by applying pressure to prepare the specimen for recording the FTIR spectra.

SEM and XRD analysis: The microstructure and morphology of *Borassus flabellifer* sheath fibers (BFSF) were visualized using a scanning electron microscope Bruker EV018 model by variable pressure method with an accelerated voltage of 22 kV and pressure level of 30 Pa. The powdered samples were subjected to XRD using Bruker Eco Advance D8 model diffractometer with monochromatic $\text{CuK}\alpha$ radiation ($\lambda = 0.15418$ nm). The generator was utilized at 40 kV and 20 mA, and the intensities were measured in the range of $10^\circ < 2\theta < 80^\circ$, typically with scan steps of 0.02° at 2 seconds per step. The integrated intensities of the Bragg's peaks in the diffractogram of BFSF were identified and their crystallinity index was calculated using the Segal's empirical method [12]:

$$\text{Crystallinity index (CI, \%)} = \frac{(I_{\text{cr}} - I_{\text{am}})}{I_{\text{cr}}} \times 100 \quad (3)$$

where I_{cr} = intensity of crystalline peak, and I_{am} = intensity of amorphous peak. The crystallite size can be calculated using the Scherer's equation:

$$L = \frac{K\lambda}{\beta \cos \theta} \quad (4)$$

where K is the Scherer's constant (0.89); β is the peak's full-width at half-maximum; and λ is the wavelength of radiation.

Thermal analysis: Thermogravimetric analysis of the fiber samples was performed using the TA Q600 instrument equipped with an auto sampler and three replications were performed. The thermograms were recorded between 20 °C and 800 °C with a heating rate of 10 °C/min in a flow of nitrogen at 100 mL/min. The TGA curves were obtained directly from the apparatus while the DTG curves were obtained by software TA advantage 5008.

Single fiber tensile strength: Single fiber tensile tests were performed on an Instron 5943 universal testing machine with a load cell of 1 kN, a cross head speed of 1 mm/min and a gauge length of 50 mm. In order to prevent grip slippage, the edge of fibers was glued between 2 mm thick polymethylmethacrylate sheets. At least 30 samples were tested and the averaged value was recorded.

RESULTS AND DISCUSSION

Moisture content: The moisture content of *Borassus flabellifer* sheath fibers (BFSF) was found as about $11.2 \pm 0.81\%$ by mass, which is comparatively higher than the other natural fibers. The higher moisture content is due to the presence of hemicellulose content, which contributes the major part of the amorphous phase in plant fiber and plays an important role in storage of moisture [13].

Chemical composition: The principal chemical compositions of BFSF are determined as cellulose, hemicelluloses and lignin of 48.20 ± 0.95 , 25.92 ± 0.83 and $23.83 \pm 0.34\%$, respectively. Cellulose content of BFSF is comparatively lesser than *Borassus flabellifer* fruit fiber, oil date palm and date palm leaf fibers but higher than coconut coir and sheath fibers [14]. The hemicellulose and lignin contents of BFSF fiber are greater than those of other natural fibers such as sisal, ramie, jute and flax fibers and this higher lignin content is responsible for microbial resistance [15].

Fiber density: The density of BFSF determined by liquid displacement test using pycnometer was found to be 0.79 ± 0.02 g/cm^3 , which is considerably lower than those of synthetic fibers and natural fibers [14]. The density of BFSF based on the chemical composition is calculated theoretically as 1.43 ± 0.03 g/cm^3 and hence the void content of BFSF fiber was found about $55.24 \pm 0.34\%$. The higher void content is responsible for the low density of BFSF, sound absorption behaviour [3] and thermal insulator [16].

FTIR studies: FTIR spectra of functional groups of BFSF show peak at 3280 cm^{-1} caused by the O-H stretching vibration and the broad band between 3600 cm^{-1} and 3100 cm^{-1} attributed to intermolecular hydrogen bonding of cellulose. The peak at 2921 cm^{-1} is the characteristic band for the aromatic C-H stretching vibration of lignin while the absorption band at 2800 cm^{-1}

can be attributed to the methoxy group of lignin [15]. The peak at 1710 cm^{-1} corresponds to the C=O stretching vibration of the carbonyl groups in hemicellulose [17]. The peak at 1612 cm^{-1} is attributed to the -OH stretching of absorbed water. The peaks at 1555 and 1525 cm^{-1} correspond to the G-ring stretching of lignin and C=C aromatic symmetric stretching, respectively [18]. The peaks at 1432 and 1370 cm^{-1} are attributed to the CH_2 symmetric bending and bending vibration of C-H of lignin [19]. The absorbance at 1318 cm^{-1} is associated to the C-O groups of cyclic ring in polysaccharides [20]. The absorbance peak at 1250 cm^{-1} represents the C-O stretching vibration of acetyl group in lignin [21]. The peak at 1150 cm^{-1} wave number corresponds to the asymmetric stretching of C-O-C linkage [22]. The absorbance peak at 1031 cm^{-1} is associated to C-O and OH stretching of polysaccharide in cellulose. The peak at 890 cm^{-1} represents the presence of β -glycosidic linkages between the monosaccharide [23].

Thermal analysis: The first derivative of thermogram (DTG) illustrated in Fig. 1, clearly revealed the inflection points. The first peak located at 51.73°C is attributed to the moisture release, the second peak at 221.74°C pertains to the disintegration of cellulose and hemicellulose and the third peak at 336.52°C corresponds to the degradation of lignin [24]. The TGA curve of BFSF shows an initial weight loss of about 11.92% between 40 and 90°C , which is attributed to the vaporization of moisture present in the BFSF fiber. The mass loss of about 2.78% between 90 and 207.9°C indicates the stability period of the fiber at the second stage. Third stage steep curve starting from 207.9°C to 304.7°C is due to the thermal degradation of hemicellulose. Fourth stage sigmoid curve between 304.7°C and 351.2°C is attributed to the degradation of cellulose. Fifth stage slow drop line from 351.2°C to 791.8°C pertains to the degradation of lignin and the molecule breaks

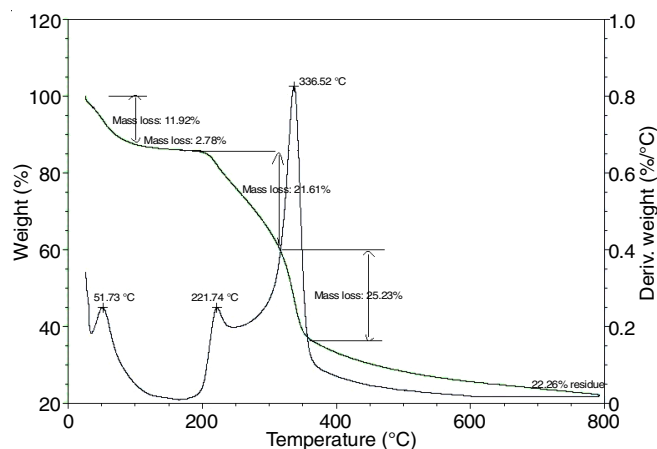


Fig. 1. TGA-DTG curve of *Borassus flabellifer* sheath fibers

down in to a variety of low molecular weight products. About 22.26% of BFSF charred residue has been obtained which is found higher those of than coir (16%) and bagasse (16%) while lower than those of banana fiber (34%) and pine apple leaf fiber (29%) [25]. A notable peak at 326°C was found similar to that of *Cissus quadrangularis* root (330°C) and *Prosposis juliflora* bark (331°C), and higher than that of coir (301°C) and bamboo (321°C) this shows the possible flinch of thermal decomposition of cellulose Ia [14].

SEM studies: Fig. 2 shows the longitudinal SEM images of BFSF which has pitting, roughness, micro-cracks on the surface thus helping in enhancing the fiber and matrix interaction. The white spots on the BFSF fiber are due to the silica crystals, which are also confirmed by SEM-EDX image (Fig. 2e). Fig. 2c-d show the cross-section of SEM images, which describes that BFSF has hollow tubular internal structure nature with polygonal shape and a central void, named lumen [17].

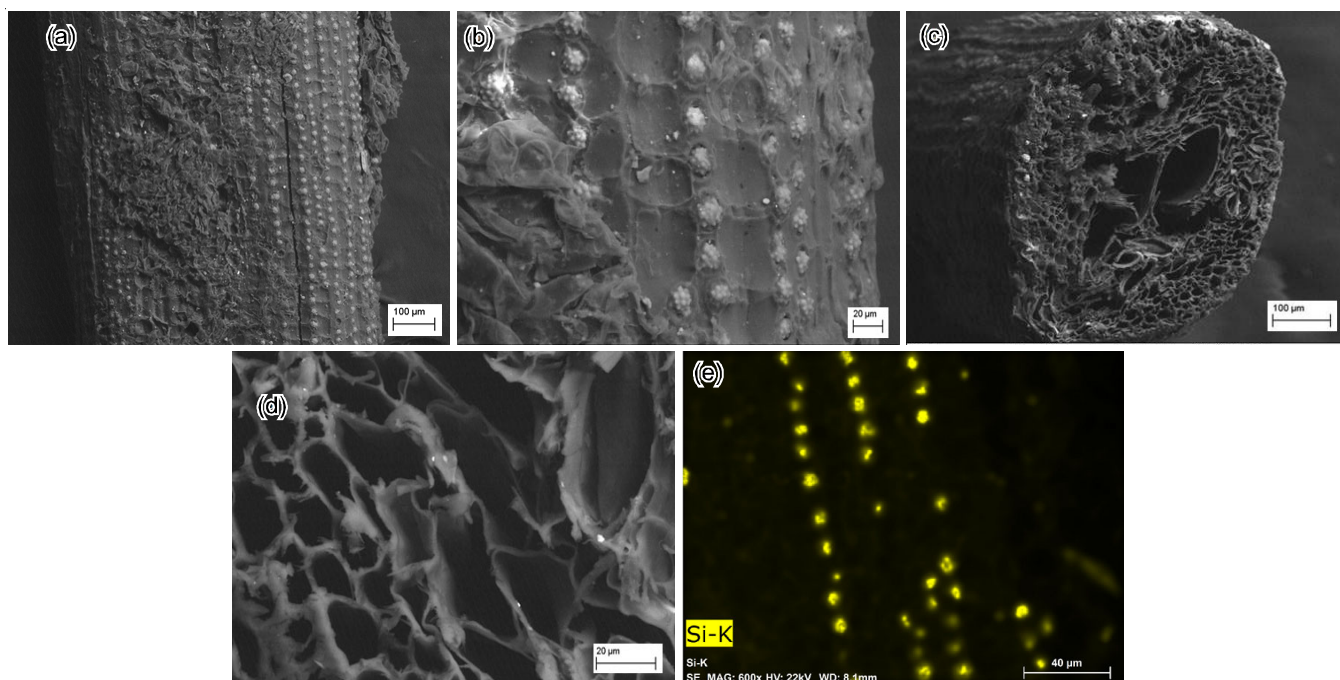


Fig. 2. SEM image of longitudinal (a and b), cross-section (c and d) view of *Borassus flabellifer* sheath fibres at two different magnifications and SEM-EDX image (e) showing the silica content of BFSF fibres

The bigger lumen size will contribute to the higher water uptake and moisture content of the fiber [26]. The larger hole, which is approximately located in the center of the BFSF, is called lacuna. The larger voids are responsible for the low density of the fiber [27,28].

XRD studies: The typical X-ray diffractogram of BFSF shows peak at $2\theta = 22.20^\circ$ reflection assigned to the (002) crystallographic plane of α -cellulose I. The CI values of BFSF were found to be 33.15%, which is lower than those of all other natural fibers but similar to that of coir and piassava leaf fibers. The lower CI value may be due to the less ordered cellulose regions [29]. The lower the CI value, the lower is the crystallinity and this affects the chemical resistance of natural fibers [18]. Using Scherer's equation, the crystallite size (L) of BFSF was found to be 2 nm, which is much lower than that determined for *R. textilis* (32 nm), ramie fiber (16 nm) and cotton (5.5 nm) but quite closer to the flax fiber (2.8 nm) [12] due to the low crystallinity with increasing amorphous domain of the fibers [30].

Single fiber tensile strength: Tensile strength of the BFSF was determined as 170-220 MPa with 10 to 22% elongation. The tensile strength of BFSF is higher than that of coconut coir sheath (46 MPa) and piassava (134-143 MPa) [15]. The higher elongation percent of 10-22% of BFSF may be due to the hollow tubular fiber internal structure, low crystallinity and high moisture content [31].

Conclusion

The characterization results clearly indicate that *Borassus flabellifer* sheath fibers (BFSF) is a better alternate material for synthetic fiber because of its lower density (0.79 g/cm^3) than glass fiber (2.5 g/cm^3), thermal stability up to 208°C and tensile strength 170-220 MPa with good elongation of 10-22%. SEM images confirmed that the fiber has textured surface topography, which enhances the fiber and matrix interaction. The higher lignin content (~24%) increases the microbial resistance and storage stability. The characteristics of high moisture, hemicellulose and void content and low crystallinity make the BFSF fiber more flexible and their morphology, low density and thermal stability are suitable for the fabrication of low-density flexible composites.

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CONFLICT OF INTEREST

The authors declare that there is no conflict of interests regarding the publication of this article.

REFERENCES

- J. Karger-Kocsis, H. Mahmood and A. Pegoretti, *Prog. Mater. Sci.*, **73**, 1 (2015); <https://doi.org/10.1016/j.pmatsci.2015.02.003>
- A. Balakrishna, D.N. Rao and A.S. Rakesh, *Comp. Part B Eng.*, **55**, 479 (2013); <https://doi.org/10.1016/j.compositesb.2013.07.006>
- P.V. Bansod, T. Mittal and A.R. Mohanty, *Acoust. Aust.*, **44**, 457 (2016); <https://doi.org/10.1007/s40857-016-0073-4>
- E. Omrani, P.L. Menezes and P.K. Rohatgi, *Eng. Sci. Technol. Inter. J.*, **19**, 717 (2016); <https://doi.org/10.1016/j.jestch.2015.10.007>
- K. Luus, *McGill J. Med.*, **10**, 121 (2007).
- T.A. Davis and D.V. Johnson, *Econ. Bot.*, **41**, 247 (1987); <https://doi.org/10.1007/BF02858972>
- P. Sudhakara, D. Jagadeesh, Y. Wang, C.V. Prasad, A.P. Devi, G. Balakrishnan, B.S. Kim and J. Song, *Carbohydr. Polym.*, **98**, 1002 (2013); <https://doi.org/10.1016/j.carbpol.2013.06.080>
- L. Boopathi, P.S. Sampath and K. Mylsamy, *Compos. Part B Eng.*, **43**, 3044 (2012); <https://doi.org/10.1016/j.compositesb.2012.05.002>
- K. Obi Reddy, C. Uma Maheswari, M. Shukla, J.I. Song and A. Varada Rajulu, *Compos. Part B Eng.*, **44**, 433 (2013); <https://doi.org/10.1016/j.compositesb.2012.04.075>
- C. Verweris, K. Georghiou, D. Danielidis, D.G. Hatzinikolaou, P. Santas, R. Santas and C. Corleti, *Bioresour. Technol.*, **98**, 296 (2007); <https://doi.org/10.1016/j.biortech.2006.01.007>
- S. Indran and R.E. Raj, *Carbohydr. Polym.*, **117**, 392 (2015); <https://doi.org/10.1016/j.carbpol.2014.09.072>
- S.S. Saravanakumar, A. Kumaravel, T. Nagarajan, P. Sudhakar and R. Baskaran, *Carbohydr. Polym.*, **92**, 1928 (2013); <https://doi.org/10.1016/j.carbpol.2012.11.064>
- A. Celino, S. Freour, F. Jacquemin and P. Casari, *Front Chem.*, **1**, 43 (2014); <https://doi.org/10.3389/fchem.2013.00043>
- S. Indran, R. Edwin Raj and V.S. Sreenivasan, *Carbohydr. Polym.*, **110**, 423 (2014); <https://doi.org/10.1016/j.carbpol.2014.04.051>
- M. Azadfar, A.H. Gao, M.V. Bule and S. Chen, *Int. J. Biol. Macromol.*, **75**, 58 (2015); <https://doi.org/10.1016/j.ijbiomac.2014.12.049>
- J. Meredith, R. Ebsworth, S.R. Coles, B.M. Wood and K. Kirwan, *Compos. Sci. Technol.*, **72**, 211 (2012); <https://doi.org/10.1016/j.compscitech.2011.11.004>
- V. Fiore, T. Scalici and A. Valenza, *Carbohydr. Polym.*, **106**, 77 (2014); <https://doi.org/10.1016/j.carbpol.2014.02.016>
- A. Bezazi, A. Belaadi, M. Bourchak, F. Scarpa and K. Boba, *Compos. Part B Eng.*, **66**, 194 (2014); <https://doi.org/10.1016/j.compositesb.2014.05.014>
- I.M. De Rosa, J.M. Kenny, M. Maniruzzaman, M. Moniruzzaman, M. Monti, D. Puglia, C. Santulli and F. Sarasin, *Compos. Sci. Technol.*, **71**, 246 (2011); <https://doi.org/10.1016/j.compscitech.2010.11.023>
- H. Yang, K. Li, X. Liu, Y. Wang, K. Liu, F. Chen, B. Deng and W. Xu, *Fibers Polymers*, **21**, 350 (2020); <https://doi.org/10.1007/s12221-020-9264-6>
- M.Z.R. Khan, S.K. Srivastava and M.K. Gupta, *J. Reinf. Plast. Compos.*, **37**, 1435 (2018); <https://doi.org/10.1177/0731684418799528>
- C. Vallo, J.M. Kenny, A. Vazquez and V.P. Cyras, *J. Compos. Mater.*, **38**, 1387 (2004); <https://doi.org/10.1177/0021998304042738>
- R. Sutrisno Soenoka, Y.S. Irawan and T.D. Widodo, *J. Southw. Jiao. Univ.*, **55**, 1 (2020); <https://doi.org/10.35741/issn.0258-2724.55.3.52>
- K. Van de Velde and E. Baetens, *Macromol. Mater. Eng.*, **286**, 342 (2001); [https://doi.org/10.1002/1439-2054\(20010601\)286:6<342::AID-MAME342>3.0.CO;2-P](https://doi.org/10.1002/1439-2054(20010601)286:6<342::AID-MAME342>3.0.CO;2-P)
- M.S. Alwani, H.P.S.A. Khalil, O. Sulaiman, M.N. Islam and R. Dungani, *BioRes.*, **9**, 218 (2014).
- L. Yusriah, S.M. Sapuan, E.S. Zainudin and M. Mariatti, *J. Clean. Prod.*, **72**, 174 (2014); <https://doi.org/10.1016/j.jclepro.2014.02.025>
- L.Q.N. Tran, T.N. Minh, C.A. Fuentes, T.T. Chi, A.W. Van Vuure and I. Verpoest, *Ind. Crops Prod.*, **65**, 437 (2015); <https://doi.org/10.1016/j.indcrop.2014.10.064>
- M. Poletto, H. Ornaghi and A. Zattera, *Mater.*, **7**, 6105 (2014); <https://doi.org/10.3390/ma7096105>
- L. Segal, J.J. Creely, A.E. Martin Jr. and C.M. Conrad, *J. Text. Res.*, **29**, 786 (1959); <https://doi.org/10.1177/004051755902901003>
- R. Moriana, F. Vilaplana, S. Karlsson and A. Ribes, *Carbohydr. Polym.*, **112**, 422 (2014); <https://doi.org/10.1016/j.carbpol.2014.06.009>
- B. Taallah, A. Guettala, S. Guettala and A. Kriker, *Constr. Build. Mater.*, **59**, 161 (2014); <https://doi.org/10.1016/j.conbuildmat.2014.02.058>