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Preparation and Characterisation of Long Natural Fibre Reinforced Thermoplastic Composites Using Direct Extrusion Compression Moulding

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Abstract:

This study explores the feasibility of a single-stage processing technique for the production of natural fiber-reinforced thermoplastic composites by integrating extrusion with compression molding. The primary objective is to streamline the manufacturing process while improving the mechanical and thermal properties of the resulting composites. Natural fibers such as jute, hemp, and flax are combined with thermoplastic matrices like polypropylene and polyethylene in a one-step extrusion process, followed immediately by compression molding. This method offers potential advantages in terms of reduced energy consumption, lower processing times, and cost-effectiveness compared to conventional multi-stage processes.

To evaluate the performance of the composites, a comprehensive analysis of mechanical properties (tensile strength, flexural strength, impact resistance), thermal properties (thermal stability, heat deflection temperature), and water absorption characteristics is conducted. Additionally, the fiber-matrix interface is optimized through surface treatments and fiber content adjustments to enhance interfacial bonding.

The results indicate that single-stage processing can produce high-performance natural fiber-reinforced thermoplastics with improved material properties, paving the way for their use in sustainable engineering applications. The findings also suggest that with proper optimization of processing parameters and fiber treatments, the composites exhibit enhanced mechanical strength, making them suitable for various industrial applications.

Keywords **— Natural Fibre, Compression Molding, Thermoplastic polymer**

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I. INTRODUCTION

The improvements of the mechanical performance of extrusion - compression moulded Natural Fibre Composite(NFC) should be possible to achieve using micromechanical analysis and tailoring the microstructure accordingly by optimizing the manufacturing processes.[1-4] The overall goals have been to find limits, regarding the material constituents and the processes and explore ways to overcome these boundaries. NFC materials processed by Extrusion-compression moulding have by default limitations regarding mechanical performance (limited fibre length, degradation at high temperatures). On the other hand NFC comprises a large variety of properties since fibres, matrices and additives can be combined in so many ways and be tailored for specific applications if a basic understanding of the micromechanical mechanisms is developed. [5-10] With these manufacturing methods a tremendous variation of products can be made. There are many products where NFC offers extremely attractive features such as large design freedom, low material cost, short cycle time, low machine wear and easy end of life handling. The market for Extrusion-compression products is huge and the environmental benefits of NFC materials over petroleum based thermoplastics are obvious.[11-12]

Attempts have clearly been done by the natural fibre reinforced thermoplastic composites for automotive sector and other semi-structural components. The trials of these types of products are already underway in Japan and other European countries, whereas such products are yet to be inducted in India. Initial efforts have been made by the Indian partner to disseminate these technology based products to some of potential users. This aspect is considered as one of the benefits for each country. The benefit of developing single stage processing of natural fibre reinforced thermoplastic composites would result in saving of the project cost and also enhances the product properties. [13-20] If single stage processing is successful, each country would greatly be benefitted in terms of production technology, cost to product ratio and more so in countless applications.

2. Materials & Methods

2.1 Chemical Treatment for Interfacial Adhesion

Natural fiber composites are also claimed to offer environmental advantages such as reduced dependence on non-renewable energy/material sources, lower pollutant emissions, lower greenhouse gas emissions, enhanced energy recovery, and end of life biodegradability of components. For this research, kenaf fiber is used instead of other natural fiber.

2.2 Alkali treatment

Alkali treatment of cellulosic fibers, also called mercerization, is the usual method to produce high quality fibers. Alkali treatment (in Figure 1) improves the fiber-matrix adhesion due to the removal of natural and artificial impurities. Moreover, alkali treatment leads to fibrillation which causes the breaking down of the composite fiber bundle into smaller fibers. In other words, alkali treatment reduces fiber diameter and thereby increases the aspect ratio. Therefore, the development of a rough surface topography and enhancement in aspect ratio offer better fiber-matrix interface adhesion and an increase in mechanical properties. Alkali treatment increases surface roughness resulting in better mechanical interlocking and the amount of cellulose exposed on the fiber surface. This increases the number of possible reaction sites and allows better fiber wetting. The possible reaction of the fiber and NaOH is as below.

Fiber-OH + NaOH ------- Fiber-O^-Na^+ + H_2O

Figure 1. Alkali Treatment of Natural fiber

Alkali treated natural fibers favoured the reinforcement in the epoxy matrix in the composite showing perfect chemical bond and better interface adhesion and thus increased the tensile strength of Hybrid composite samples. The failure of Natural fiber–epoxy Hybrid samples, characterized by brittle failure, showed long tails after the predominant damage. It is thus estimated that an interfacial interaction in the present composite would result in a higher elongation to break due to alkali treatment. We can clearly absorb the fiber wetting of the treated fiber and also good fiber matrix interaction.

The weight loss was calculated from the following equation:

Weight loss = $W_0 - W_1/W_0 \times 100\%$

Where w_0 denotes the weight of sisal fibers before NaOH treatment, and $w_1$the weight of fibers after having been treated with NaOH.

2.3 Silane Treatment

The distilled water were taken and kept in a separate container's for one hour. The silane treatment has been done on fibers by that which provides fiber matrix adhesion to stable properties of composite. The sodium chloride pellets were dropped in a container which consists of water by that the 5% concentration of the silane solution has been prepared. The fibers were soaked separately in a silane solution and allowed to soak 2 hours in the solution. The fibers were taken out from the container after two hours and again were washed with distilled water. These fibers were kept under sunlight for 1 week and dried.

Silane is used as coupling agents to modify fibre surface. It undergoes several stages of hydrolysis, condensation and bond formation during the treatment process with the fibre. Silanols forms in the presence of moisture and hydrolysable alkoxy groups. It reacts with cellulose hydroxyl group of the fibre and improves fibre matrix adhesion to stabilize composite properties. The chemical composition of silane coupling agents (bifunctional siloxane molecules) allows forming a chemical link between the surface of the cellulose fibre and the resin through a siloxane bridge. This co-reactivity provides molecular continuity across the interface region of the composite. It also provides the hydrocarbon chain that restrains fibre swelling into the matrix.

Drying the fibers at room temperature | Drying of fibers in oven | NaCl treatment for silane treatments

Figure 2: Silane treatment

Natural fibres exhibit micropores on their surfaces and silane coupling agent act as a surface coating which penetrates into the pores and develop mechanically interlocked coating on their surface. Silane treated fibre reinforced composite provides better tensile strength properties than the alkaline treated fibre composites. the effect of alkali (5% NaOH for 2hrs) and silane (1% oligomeric siloxane with 96% alcohol solution for 1 hr) treatment on the flexural properties of jute epoxy and jute polyester composites. For jute epoxy composites alkali over silane treatment (Figure 2) resulted in about 12% and 7% higher strength and modulus properties compared to the alkali treatment alone.

The mechanical properties of long natural fibre reinforced thermoplastics depend mainly on natural fibre distribution; viz. Fibre length distribution and the degree of wetting. By changing Screw configuration and Screw speed, test specimens were generated with varying degree of fibre distribution by direct in-line compression moulding. Tensile and Flexural strength of such specimens are analyzed with respect to fibre distribution. These results are useful to set process parameters for extrusion process and to set targets for product characterization.

The Bi-lobed co-rotating twin screw extruder is the industry standard for long fibre compounding. The screws are termed ‘self-wiping’, and the material follows the form of the number ‘8’ pattern. There are infinite numbers of screw design variations possible. However, there are only three basic types of screw elements which are possible. Depending on extruder designers the designation of the elements differs.

In the present study, the sankhla screw elements that used are RKB, 3KB and SME. Special screw elements (OSE) and special mixing elements (OME) are also used in designing the specific screw configuration to have optimum fibre length in the extrudate.

The materials used for the first two trials are homo polymer Polypropylene and Sisal fiber. The homo polymer Polypropylene is used as a matrix, which is in granular form (Repol H11UMA – MFI 11.0 grams/10min, Density 0.902grams/cm³). Sisal fiber (fiber diameter 50µ) is in the form of continuous roving. Sisal fiber used in the experiment is specially designed for the manufacture of long fibre thermoplastic composites. To have a better coupling between the fibres and the matrix 2.0% of grafted polypropylene is used as a coupling agent (OPTIM P408 Density 0.91grams/ml) and 0.1% of thermal antioxidant (AO -1010) was dry blended using high speed mixture with the homopolymer Polypropylene.

2.3.1 Process Setup

The compounded extrudate of the LFRTP composite were produced with extrusion process followed by pressing using hydraulic press considered to be a single stage processing of long fibre reinforced thermoplastic composite (LFRTP-D) process. The schematic representation of the experimental set up is shown in Figure 3.

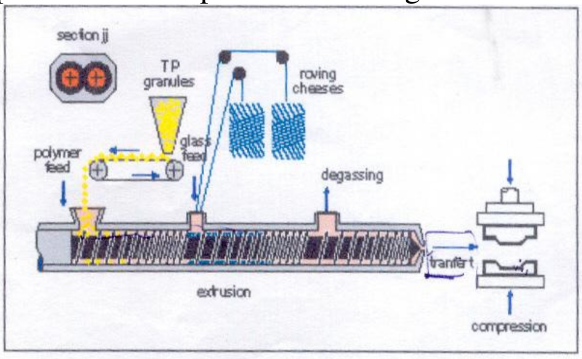


Figure 3 Schematic representation of the LFRTP direct process

The extruder used is a modular, supreme process capacity with deeper flights OMEGA-40, SANKHLA, Generation Next Intermeshing Co-Rotating Twin Screw Extruder (L/D 48:1, Do/Di 1.71 and specific Torque ≥ 11.3 N-m/cm³). The moulded specimens were produced using 50T Hydraulic press, which is considered as compression moulding. The matrices were melted in such way that the molten matrices are free from solid molten interfaces and voids before introducing the natural fiber roving. The natural fiber is preheated before introducing into extruder. After introducing the natural fiber roving into the extruder, the compounded rectangular shape extrudate, with predefined dimension was cut and collected at die manually and immediately transport to the hydraulic press. The required hydraulic pressure was maintained to mould the LFRTP composite plate.

In first trial screw configuration is designed using screw mixing element (SME), Bi-lobed forward kneading block (RKB) and tri-lobed eccentric kneading block (3KB). The positions of these elements are six times the screw diameter before from screw end. The varied parameters used for studying the influence of screw elements are screw speed (100RPM and 200RPM) and fibre concentration (30wt%). The mould dimension used to produce the LFRTP specimens is 210mm x 160 x 4mm. The mechanical properties were analyzed by using ISO standards. The tensile properties are studied as per ISO 527. The specimens are cut and tests are performed as per the standards.

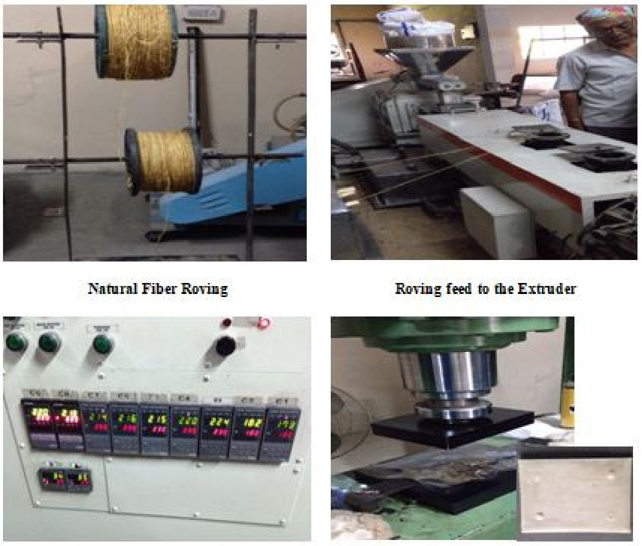
For the trials an OMEGA-40 co-rotating twin screw extruder (Sankhla polymers) was used. The OMEGA extruder series is known for an extreme high $^{Do}{}_{Di}$ ratio ($^{Do}{}_{Di}$=1.71) that offers best feeding ability, highest speed of operation and output rates. To ascertain the effects of the individual elements only 6D length the barrel was used.

Twin Screw Extruder Design For Long Fiber Processing

Figure 4 NR II –S G Twin Screw Extruder

In the plastics industry, there are three main extruder types: the screw extruder, which is the most common, the ram extruder, and the drum or disk extruder, which is the least common. In a screw extruder, a screw rotates in a cylinder; the rotation of the screw creates a pumping action.

Twin-screw extruders (Figure 4) can run at high or low speed, depending on the application. High-speed extruders run at approximately 200-500 rpm or higher; they are primarily used in compounding. Low speed extruders run at approximately 10-40 rpm and are used mostly in profile extrusion applications. Most twin-screw extruders for profile extrusion are counter-rotating extruders. This is because counter-rotating extruders tend to have better conveying characteristics than co-rotating extruders. Most twin screw extruders have parallel screws, but some extruders have conical screws where the screws are not parallel. Another distinguishing feature of twin extruders is the extent that the screws intermesh. The screws can be fully intermeshing, partially intermeshing or non-intermeshing. Most twin-screw extruders are intermeshing. The advantage of non-intermeshing extruders is that they can have a very long length without problems with respect to metal-to metal contact between the screws. The length to diameter ratio (L/D) can be100:1 or higher. The L/D of intermeshing twin-screw extruders is generally limited to values less than 50:1. A disadvantage of current non-intermeshing twin screws is that they have limited dispersive mixing capability. Mixing takes place both in the melting zone as well as in the melt conveying zone of the extruder. The solid plastic typically moves in plug flow, which means that there is no relative motion between the solid plastic particles. Multiple extruder apparatus for compounding thermoplastic resin and reinforcing fibers incorporates a resin extruder in which thermoplastic resin are melted and a compounding extruder in which the molten thermoplastic resin is mixed in intimate contact with long reinforcing fibers of at least one inch in length. The melted thermoplastic resin is introduced into the compounding extruder at a point downstream of the inlet point for the reinforcing fibers, so that the fibers are mechanically worked and heated before coming into contact with heated, molten thermoplastic resin.



Natural Fiber Roving

Roving feed to the Extruder

Temperature reading at barrel zones

Extruder has been pressed by using hydraulic press to obtain desired shape & Inset image (Long sisal fiber reinforced thermoplastic composite)

Figure 5 Fabrication of Specimen

3. Result and Discussion

3.1 Testing Of Specimens For Mechanical Properties

Mechanical properties such as tensile strength, tensile modulus, flexural strength were tested according to the ASTM standards.

The obtained hybrid composite plates were cutted according to ASTM composite standards for the characterizations. Some of the tests were conducted in order to characterize the mechanical properties.the testing was carried out by the computer integrated universal testing machine (UTM) which has the capacity of 100KN Kalpak software is used for the data acquisition the testing.

3.2 Tensile Test

The desired dimension of specimen for mechanical testing was cut by the fabricated composite. Tensile strength of a material is maximum amount of tensile stress that it can be taken before failure. Dog-bone type specimen (Figure 6) is the commonly used specimen for tensile test. According to the ASTM D-3039, the tensile specimen was made and the dimension are shown in the below table.

Calculation for Tensile Strength

Tensile Strength = W/(b×d)

Where, W is the ultimate failure load (N)

b is mean width of sample (mm)

d is mean thickness of sample (mm)

Tensile Strength = W/(b×d)

= 645/ (25×3)= 8.60 MPa

Figure 6 Dog Bone Shape for Tensile Test Specimen

Table 1 Dimension for Tensile Test Specimen

Sl. No.	Specimen composition	Peak load (N)	Ultimate tensile strength (Mpa)
1	20%	436	9.08
2	30%	639	14.15

Total length in (mm)	Span length in Mm	Width in mm	Thickness in mm
250	170	25	3

Then the prepared specimen with the ASTM D-3039 dimension was taken and the specimen was taken for testing.

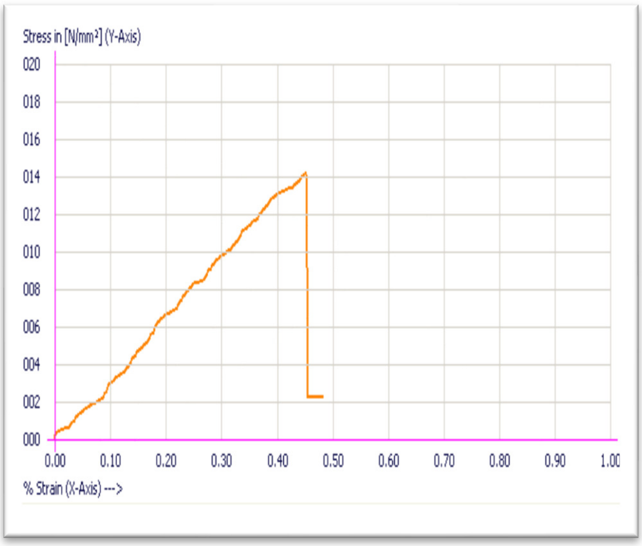


Figure 7 Graph of Stress V/S Strain for The Tensile Test

The above graph (Figure 7) shows the results for stress v/s strain in tensile test where the maximum stress is 14.15 Mpa and the Strain is 0.48 for one of the tensile test which was carried out.

Figure 8 Breakage of Tensile Specimen

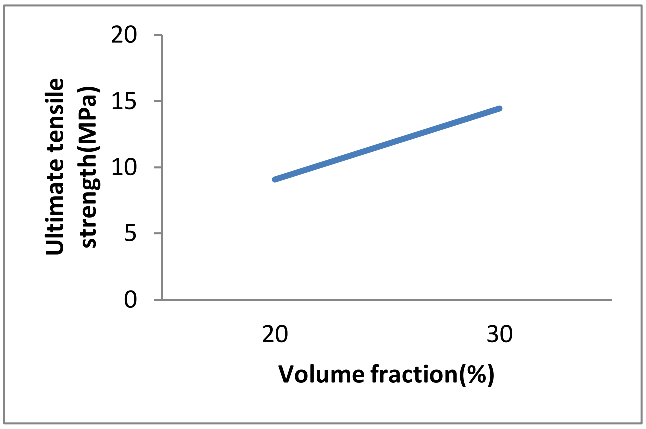
In the above figure 8 after the testing of the dog-bone shape tensile specimen we have showed that the breakage of the tensile specimen. In the figure the first specimen was 20% volume fraction specimen where the specimen broke at the tip of the neck and the second shows the 30% volume fraction specimen where it as broke with maximum ultimate tensile strength by comparing to the 20% of volume fraction specimen. Also by comparing the experimental and theoretical we got the percentage of error is less than 10%

Table 3 Tensile Test Results

Figure 9 Graph of Ultimate Tensile Strength v/s Volume Fraction

The above graph (Figure 9) shows the result of the ultimate tensile strength v/s volume fraction of the hybrid composite specimen where the ultimate tensile strength for 20% of volume fraction is less compare to the volume fraction of 30%.

3.3 Flexural Bending Test (3 Point Bending Test)



The second test which we carried out was the three point bending test that is the flexural test specimen (Figure 10) this was chosen because it requires less material and eliminates the need to accurately determine center point deflections with test equipment. According to the ASTM D-790 test was carried out and the dimensions are given below.

Calculation for Flexural Strength

$$\sigma = 3PL / 2bh^2$$

Where,

σ = stress at the outer surface of mid span (Mpa).

P = Applied Force (N).

L = Support Span (mm).

b = Width of the beam (mm).

h = Thickness of Beam (mm).

$\sigma = 3PL / 2bh^2$

$\sigma = (3\times46\times80) / (2\times12.5\times3^2)$

σ = 49.06 Mpa.

Figure 10 Flexural Bending Test Specimen

Table 4 Dimension for Flexural Bending Test

Total length in (mm)	Span length in (mm)	Width in (mm)	Thickness in (mm)
125	100	12.5	3

The above specimen considering the ASTM D-790 was taken and kept in the UTM machine (Figure 11) and the load was applied at the center point.

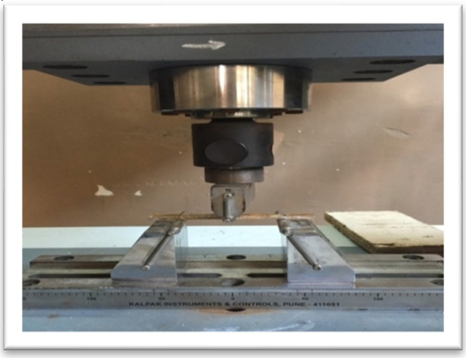


Figure 11 Flexural Bending Specimen Kept in UTM

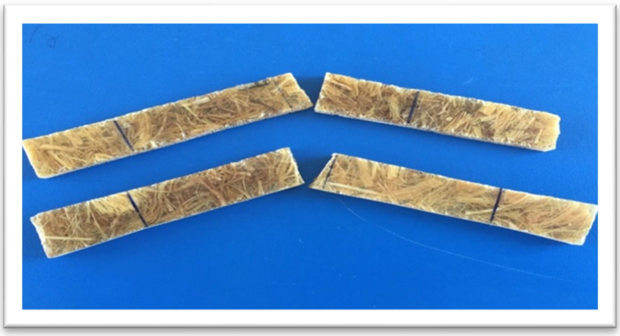


Figure 12 Breakage of Flexural Bending Tested Specimen

Table 5 Flexural Bending Test Results

Sl. No.	Specimen composition	Peak load (N)	3 point flexural strength (Mpa)
1	20%	46	48.94
2	30%	76	81.41

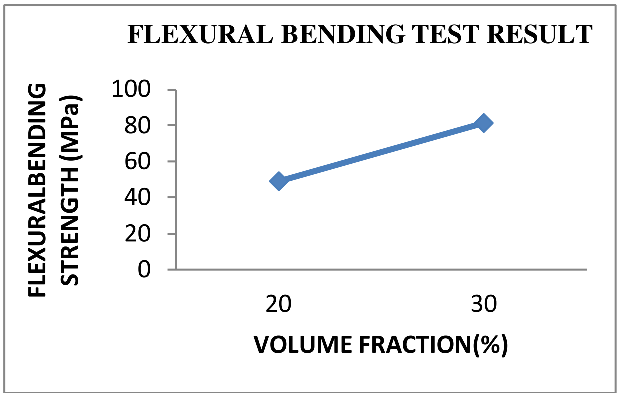


Figure 13 Graph of Flexural Strength v/s Volume Fraction

The second test which was conducted on the hybrid composite plate (Figure 12) was the flexural test. In this test for each volume fraction we conducted two tests and the better results was shown. For the 20% of volume fraction the flexural bending strength was 48.94 and for the 30% of volume fraction the flexural bending strength was 81.41 as shown in the above graph (Figure 13). When the percentage of volume fraction is increased the better results were obtained in the above two tests. Also the theoretical test was calculated and by observing both experimental and theoretical results for flexural test the percentage of error was calculated and it was less than 10%

3.4 Hardness Test

For hardness test a dimension of 80*60*3 mm specimen (Figure 14) was cut by the fabricated composite. Hardness is defined as the ability to oppose to indentation, which is obtained by measuring the stable depth of the indentation. Test was calculated by the Brinell Hardness test machine (Figure 5). In this we can find the Brinell hardness number.

Figure 14 Hardness Test Specimen

Figure 15 Brinell hardness Testing Machine

Table 6 Hardness Test Results

Sl. No.	Specimen composition	Load in kg	Scale	Average BHN
1	20%	100	B	53
2	30%	100	B	76

In this hardness test the indentation diameter is 10mm and the load applied was 100kg. The load was applied at 3 different points in the specimen and the average of that was taken as final reading. The obtained average Brinell hardness number was for 20% volume fraction it is 53BHN and for 30% volume fraction 76 BHN.

4. Conclusion

The study demonstrates the potential of single-stage processing for the production of natural fiber-reinforced thermoplastic composites, successfully integrating extrusion with compression molding. This streamlined method offers significant advantages in terms of reduced processing time, energy savings, and cost-effectiveness compared to traditional multi-stage techniques. The resulting composites exhibit improved mechanical properties, including enhanced tensile strength, flexural strength, and impact resistance, along with notable thermal stability and moisture resistance. Optimization of fiber-matrix interaction, through surface treatments and fiber content adjustments, proved critical in achieving better adhesion and dispersion of fibers within the thermoplastic matrix. Characterization techniques, such as dynamic mechanical analysis (DMA), provided valuable insights in the performance under various conditions.

Overall, single-stage processing presents a promising approach for manufacturing sustainable, high-performance natural fiber-reinforced thermoplastics. These findings suggest that, with further refinement, the process can be applied to a range of industrial applications, promoting the use of eco-friendly materials in engineering and construction. Future research could focus on optimizing specific parameters to further enhance the performance and durability of these composites.

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