



## Strength shear test for adhesive joints between dissimilar materials obtained by multicomponent injection

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### ARTICLE INFO

#### Keywords:

B. Composite  
C. Lap-shear  
C. Stress analysis  
D. Hybrid joints polymer adhesion

### ABSTRACT

The multicomponent injection molding process enables the combination of different materials in a single step. However, one of the challenges of this new technology is related to the adhesive strength between dissimilar materials. In this work, specimens were overmolded with dissimilar composite materials made of polypropylene and coconut fibers added with coupling agents. By means of a new patented device specially produced to evaluate the adhesive strength of such different materials under pure shear stress, it was possible to measure the force necessary to promote the detachment of dissimilar surfaces. Electron microscopy and contact angle analyses were used to better understand the adhesion phenomenon between such dissimilar materials. Although a maleic anhydride additive promoted better anchoring between fibers and the polymer matrix, it was observed that the composite without coupling agents exhibited the greatest adhesive strength between dissimilar surfaces.

### 1. Introduction

Natural fibers, or lignocellulosic fibers, have been widely used as reinforcements in composites, mainly because these materials are cheap, with low density, biodegradable and are from renewable resources, as pointed by Saheb and Jog [1]. However, plant fibers are quite hydrophilic, which results in poor chemical interaction with polymers, which are usually hydrophobic. To minimize this problem, physical and chemical processes have been used to modify fiber surfaces to improve matrix-fiber interfacial adhesion as observed by Xie et al. [2]. Lu et al. [3] verified that the incorporation of polar natural fibers into nonpolar polyolefin thermoplastics requires the presence of coupling agents (CAs) to obtain better adhesion conditions. As compatibilizers have functional groups that promote polar adhesions in such new materials, they react chemically with the hydroxyl present in the fiber through strong covalent bonds (or secondary acid-base interactions or even hydrogen bonds).

The development of composite materials applicable for injection molding represents one of the many possibilities of combining multicomponent materials for designs of plastic parts, as Budhe et al. [4] mentioned in their research. Nowadays, there is a growing interest to optimize the strength, weight, and durability of structures with composite materials. Therefore, it is necessary to better understand the phenomena between bonding of dissimilar materials in order to obtain

the benefits provided by multi-material joints [4]. Consequently, an appropriated combination of overmolded materials might be the key to develop lighter, strong and eco-friendly structures.

Multicomponent injection is being increasingly used in the current production of plastics because it allows the combination of different materials in a single process, which in turn allows the manufacture of parts with a bold design, of higher added values and shorter manufacturing times, as pointed by Priyadarshi et al. [5]. Overmolded parts made up of dissimilar materials, with a component that is made by a composite containing natural fibers, is an ecologically viable option with new possibilities for innovation. The procedure uses multiple molds where the first material is injected by means of a single material molding (SMM), then the mold rotates 180° around its own axis and receives a second material in few seconds to combine with the previous. However, the presence of an already-molded component during the second phase (or the use of subsequent molding phases) means that this technology differs from the traditional injection molding process, as mentioned previously. According to Banerjee et al. [6], the manufacturing problems may occur for many reasons, including material incompatibility, the location of injection points, polymer moldability and other problems due to the ejection system. Another factors of fundamental importance for a better adhesion between polymers are related to the influence of some process variables, as plastification, polymer injection, time processing, pressure, temperature gradient and

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<https://doi.org/10.1016/j.ijadhadh.2018.08.007>

Accepted 8 August 2018

Available online 23 August 2018

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cooling, during each one of the overmolding stages, as explained by Piotter [7]. These variables, when adjusted properly, will guarantee the quality of the resulting material and the reproducibility of the process.

To obtain good adhesion, the bonds between two polymers must be known, which can be considered the sum of physical (mainly mechanical) and chemical forces that overlap and influence each other. Da Silva et al. [8] reported that the mechanical forces act on porous and/or roughness surfaces improving anchoring of dissimilar materials, providing better adhesion. The performance of adhesively bonded joints depends on many parameters such as surface preparation, composite interfaces, chemical bond characteristics between adhesive and adherend parts, overlap length, among others. However, this adhesion depends on a good adhesive wetting on the adherent surface injected on the first stages.

According to Packham [9], there is intimate contact between adherent and adhesive materials for best results, which is related to the surface wetting properties of dissimilar materials. The degree of wetting (wettability) of a surface is of crucial importance for better adhesive joints between polymers. The *Surface Free Energy* (SFE) is a physico-chemical property of a surface that can be determined indirectly by wettability measurements, as pointed by Kraus [10]. To quantify this wettability, the measurement of the contact angle of a drop with the surface of the material at the triple point of contact (between solid, liquid and vapor phases) is important. This measure is related to the "wetting surface energy" characterized by the Young and Dupré Eq. (1):

$$\gamma_{sv} = \gamma_{sl} + \gamma_{lv} \cos\theta \quad (1)$$

where  $\gamma_{sv}$  is the surface tension between solid and vapor phases,  $\gamma_{sl}$  is the surface tension between solid and liquid phases, and  $\gamma_{lv}$  is the surface tension between liquid and vapor phases.

According to Bracco and Holst [11], by measuring the contact angle, certain considerations may be observed. For surfaces in which the contact angle is less than 90°, the material is classified as hydrophilic, and for an angle greater than 90°, it is hydrophobic. Thus, when more hydrophobic is the surface, worse is its wettability, and consequently its adhesion ability.

Other adhesion mechanisms consist of electrostatic forces arising from the difference in electronegativity between materials. Forces of chemical origin are present as primary (ionic, covalent) or secondary (Van der Waals or hydrogen-based) interatomic bonds, which depend on the binding energy between atoms, as explained by Devries and Adams [12]. The mechanism that best represents adhesion in polymers is the adsorption theory. It assumes that adhesion results from atomic bonding on material surfaces. Van der Waals forces are the most common in terms of adhesion, but there can be hydrogen-bond and acid-base interactions, which can also influence adhesion [9].

There are several methods used to test the adhesive strength of polymeric materials and a commonly used standard for evaluating adhesive strength is the "T-Peel Test" – according to ASTM D1876 Standard [13]. Additionally, there are many peel test methods. It is possible to cite quite a few: the Floating roller (or without rollers) moving table; Floating roller (115 degrees); Peel wheel and 180-degree Peel [10], among others. All the mentioned tests focus on measuring the force required to detach two surfaces by a peeling stress procedure, without assessing the force required to displace one surface over the other by shear stress. Therefore, some researchers as Li et al. [14], proposed several techniques for reducing peel and interfacial stresses as: spew fillet, adhesive thickness, mixed adhesive, tapered plate with different shapes, different thickness, adherent widths, tapered length and thickness, etc. When the polymers are rigid and plane the Lap Shear Test" (Strength of Adhesively Bonded Rigid Plastic Lap-Shear Joints in Shear by Tension Loading - ASTM D3163-01) [15] could be adopted.

However, all of the above tests focus on measuring the force required to separate two surfaces. They don't assess the pure shear stress due to the presence of other forces as those generated by the bending moment. The single lap joint geometric aspect favors the generation of

bending moments, since the tensile stress T is converted into T' due to sample misalignment, as explained by Bamberg et al. [16].

As a means to evaluate the adhesive strength between dissimilar polymers and composites, in this work a specific procedure was applied using a granted patent of a new mechanical device as described previously by the present authors (BR 10 20160 21054) [17]. This device measures the adhesive strength between two overmolded materials in an overlap region, disregarding the influence of forces resulting caused by bending moment resulting in peeling stress. To test and present the results using this new device, it was used for evaluation of the influence of coconut fibers and the interaction of coupling agents over adhesive forces for overmolded polypropylene and fiber composites.

## 2. Materials and methods

In order to analyze the influence of the coupling agents on adhesive strength, the samples were injected *in situ* and subjected to a new tensile test. To evaluate the adhesive strength of the overmolded samples it was used a specific granted device proposed for this study (BR 10 20160 21054). More specifically, surface and morphological analysis was done by means of scanning electron microscopy (SEM). As complementary investigations, a contact angle test and optical microscopy of the surface region were performed to correlate these results with bond forces between dissimilar surfaces.

### 2.1. Selection and preparation of composites

To evaluate the influence of the coupling agents on the adhesion strength between dissimilar materials (copolymer matrix and coconut fiber) four composite formulations mixed with dry coconut fibers were developed. The selected polypropylene was EP 440L (Braskem), an ethylene heterophasic copolymer with flow rate of 6.0 g/10 min. The coupling agents used were: i) Orevac CA 100, a polypropylene (PP) grafted with 1wt% maleic anhydride content; ii) Orevac 18507, a high density polyethylene (HDPE) also grafted with 1 wt% maleic anhydride (both supplied by Arkema Company); iii) Cesa® Mix PEA0601031, from Clariant Company, which consists of titanate-functionalized polyethylene that uses stearamine as a lubricant. This last coupling agent was customized for a specific application in composites with natural fibers to the SENAI CIMATEC. In this study, coupling additives with PE and PP were tested to verify which has the best interaction between the polymer matrix and coconut fibers. Polypropylene and polyethylene are immiscible and partially compatible [14], and these blends have been attracting special attention due to their potential industrial applications. One of the reasons for this specific study is related to an improvement in its mechanical properties, more specifically on impact tests, especially at low temperatures, and environmental stress cracking properties. Due to immiscibility, in order to enhance the ultimate properties, a compatibilizer such as ethylene-propylene rubber (EPR), already contained in the PP EP 440L, is frequently used to improve mechanical properties such as the tensile impact strength reported by Kesavan [18] and Utracki [19].

All these coupling agents showed compatibility with the materials used in this work, as shown in Table 1. In the preliminary investigation, the objective was to identify the best compatibilization compositions to attain maximum improvement in ultimate mechanical properties of the resulting overmolded materials.

Each formulation was premixed for 2 min using tumbling process, and the formulation was dosed into the main feed of an Imacon co-rotating twin screw extruder, model DRC 30:40, with a thread diameter of 30 mm and L/D aspect ratio of 40 and under 190 °C melt temperature. The thread profile that was used in the process is the most commonly applied one for the composite types used in such works. The contents of coconut fiber and the coupling agents presented in Table 1 are relative to the total mass of the composite sample. The coupling agent corresponds to 6 wt% of fiber mass.

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