

Acidic whey as a novel coupling agent for composites based on E-Glass fibers and low-density polyethylene#

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Abstract

In order to enhance the interfacial-adhesion between short E-glass fibers and low-density polyethylene (LDPE), acidic whey; a dairy byproduct was applied as coupling agent. The fibers were immersed in acidic whey for 12, 24, 36, 48 and 60 hours. Composite materials containing 20 wt.% E- glass fibers and LDPE were prepared by extrusion process. The tensile strength and modulus of the produced composites were improved by using acidic whey, and the improvement in those properties was strongly affected by the immersion time of E-glass fibers in acidic whey. An increase of about 289%, 107%, 291% and 721% in modulus of elasticity, tensile strength, modulus efficiency and strength efficiency factors respectively were achieved after 60 hours of immersion of glass fibers in acidic whey. The effects of acidic whey on tensile properties and efficiency factors could be ascribed to the mend in interfacial-adhesion between glass fibers and LDPE polymer.

Keywords: Acidic Whey, Low Density Polyethylene, Glass Fiber, Strength Efficiency, Modulus.

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Introduction

Mechanical properties of fiber-reinforced plastics are highly ruled by the interfacial-adhesion between the polymeric matrix and the reinforcement. The applied load is transferred from the polymer via the interface to the reinforcement fibers, therefore, poor interfacial adhesion affects adversely the performance of composite materials because of ineffective load transfer between polymeric matrix and reinforcements. Chemical treatments such as coupling agents, acetylation, and alkalization were used to promote the interfacial-adhesion and create an effective transfer of load between composite constituents. (H. H. Kim, S. Y. Kim, D. H. Kim, Oh & Jo, 2013, p. 38; Muhammad & Ahmad, 2013, p. 612; Kumar, Obrai & Sharma, 2011, p. 219; Kumar, Chandrasekaran, Santhanam & Sudharasan, 2017, p. 1; Poletto, 2017, p. 433; Dung, The & Kotek, 2016, p. 300; Kim, *et al.* 2016, p. 79).

Many coupling agents such as silane, polyethylene-grafted maleic anhydride (PE-g-MA), polypropylene-grafted maleic anhydride (PP-g-MA) and styrene-ethylene/butyl-diene styrene-grafted maleic anhydride (SEBS-g-MA) were utilized to improve thermal and mechanical properties of the glass reinforced plastics (Sair, *et al.* 2017, p. 550; Roumeli, *et al.* 2015, p. 93; Venkatachalam, Navaneethakrishnan, Rajsekar & Shankar, 2016, p. 555; Raj, Zhou & Fan, 2018, p. 266; Kumar, Obrai & Sharma, 2011, p. 219; Awanis, Sofia & Samat, 2012, p. 390; Simão, *et al.* 2016, p. 746).

The use of epoxy silane improved the impact and ductility of polylactic acid-wood flour-based composites due to the enhancement of the interactions between constituents of the composite material (Wang, Qi, Xiong & Huang, 2011, p. 281), while the addition of octanoic acid and PP-g-MA was also found to promote strong interfacial adhesion recycled polypropylene and wood flour which reduced the expansion coefficient of the produced composites (Poletto, 2018, p. 563).

An increase of 23% and 39% in modulus and tensile strength respectively of hemp-polyurethane composites was observed because of the use of alkaline and silane treatments (Sair, *et al.* 2017, p. 550). The use

of (PE-g-MA) as a coupling agent created a strong interaction between high-density polyethylene hemp fibers and caused an improvement of composite performance and strength (Roumeli, *et al.* 2015, p. 93). An addition of 8 wt% of (PP-g-MA) increased tensile, flexural and impact strengths of polypropylene-short glass fibers composites with 18%, 24% and 33% respectively (Lin, *et al.* 2015, p. 8279). Furthermore, 4 wt% of 3-glycidoxypolytrimethoxysilane enhanced tensile, flexural and impact strengths of 30 wt% basalt fiber-nylon based composite by 42%, 37% and 223%, respectively (Deák, Czigány, Tamás & Németh, 2010, 590).

Rao *et al.* studied the effect of coupling agent on the compatibility and interfacial interaction between wood flour and polyethylene by the use of Fourier Transform Infrared (FTIR) and ¹³C Nuclear Magnetic Resonance spectroscopy (NMR) which revealed the formation of chemical reactions between the composite components contributing directly in the enhancement of compatibility and interfacial adhesion (Rao, Zhou & Fan, 2018, p.266). Moreover, Natural gums were employed as compatibilizers for biodegradable polymer blends and as coupling agents for polymers reinforced with cellulosic fibers (Inga-Lafebre, *et al.* 2019, p.296).

Whey is the liquid left over from the manufacturing of dairy and casein. Whey is a byproduct of the dairy industry that accounts for 80-90 percent of total milk volume. About half of the components in the original milk, including as proteins, lactose, vitamins, and minerals, are found in this residue. Whey is divided into two types: sweet whey, which has a pH of 5.9-6.6, and acid whey, which has a pH of 1.1-4.5 (Lievore, *et al.* 2015, p. 2083). If acidic whey is emitted into river systems, it may contribute to the organic pollution of the environment and it has the potential to deplete water oxygen levels (Flinois, Dando & Padilla-Zakour, 2019, p. 7874). Therefore, whey has been utilized for food and biotechnological applications to overcome or minimize its environmental impact. Some valuable products such as whey protein, lactic acid, functional foods and beverages, bioethanol, biogas, bioplastics, etc. are extracted or developed from whey (Zandona, Blažić & Jambrak, 2021, p.147). But the need for refining technologies and integrated processes to convert whey to such products have directed and encouraged scientists and researchers to look

for utilization the whey with minimum efforts and without pretreatment steps in other direct applications. Therefore, the use of acidic whey as a coupling agent in polymer/fiber composites would be of scientists and engineers' interest due to its low cost, abundance, versatile properties and ease of processing in addition to the ability of scaling up the process due to the simplicity and the lack of sophisticated required techniques as will be illustrated in experimental section.

This work aims to utilize acidic whey for the first time (to the best of our knowledge) as a natural coupling agent to improve the interfacial-adhesion between E-glass fiber and LDPE and then to investigate the effect of immersion times of E- glass fibers in acidic whey on the tensile properties of the prepared composites.

Experimental Part

Materials

Low-density polyethylene (LDPE) (IPethen ®323), was purchased from Carmel olefins LTD, with a flow index of 2 g/10 min @ (2.16Kg /190 °C) and a density of 920 kg/m³, was used as a polymeric matrix to produce composite materials. Short E-glass fibers having a length of 38.8 mm and 10.6 µm in diameter, supplied by Vosschemie, were used to reinforce LDPE. The tensile strength, modulus and the density of E-glass fibers were 3.45 GPa, 72.4 GPa and 2540 kg/m³, respectively.

Acidic whey collected from local dairy plant was filtered to remove contaminants and impurities, then stored for further use.

Preparation of Composite Materials

E- glass fibers were immersed in small volume of acidic whey for 12, 24, 36, 48 and 60 hours at room temperature to investigate the influence of acidic whey treatment on the composites' tensile properties. The amount of whey used in immersion process was very little to wet only the fibers and in order to be evaporated completely during the drying process. To remove all moisture (remaining liquid whey), the treated fibers were dried in a drying oven at 120 °C for 4 hours. Then fibers were cut manually into small short fibers having an average length of 8 mm. 20 wt.% of treated

short fibers and LDPE were blended together by using a simple tumbling apparatus. The blend was extruded at 200°C at a speed of rotation of 17 rpm by using a homemade single-screw extruder with length (L) to diameter (D) ratio of 20 and 6mm rod die. The preparation procedure is summarized in schematic 1. Herein, the authors would like to declare that the proposed preparation protocol is reproducible according to outputs and findings illustrated in next sections.



Schematic (1): Low density polyethylene/glass fiber composites preparation.

Testing Methods

To investigate the effect of the extrusion process on the residual (remaining) fibers content in the polymeric matrix, three samples of each prepared composite were placed in a furnace at 450 °C for 5 hours to burn LDPE matrix, and then the mass of the remaining fibers was measured to estimate the residual fibers content.

Gunt Hamburg apparatus WP310 was used to conduct tensile test, the test was carried out at a constant speed 5 mm/min. For each prepared

composite, five samples of 130 mm gauge length and 6 mm diameter were tested. The test was carried out according to ASTM D638-14 standard.

Results and Discussion

Residual Fibers

Table (1): Residual fiber content for the produced composites.

Treatment time (hours)	Residual fibers wt.%
0	10.52 \pm 0.35
12	10.50 \pm 0.28
24	10.55 \pm 0.31
36	10.83 \pm 0.39
48	11.61 \pm 0.23
60	12.07 \pm 0.42

A residual fiber test was performed to explore the effect of extrusion process on the fiber percent in the polymeric matrix. The residual fiber contents of prepared composite are shown in Table 1 for different immersion times. It can be observed that the residual fiber mass varies from 10.50 \pm 0.35 to 12.07 \pm 0.42 wt.%. The drop in fiber content can be attributed to the fiber sticking in the extruder zones and/or the extrusion hopper.

Tensile Properties

Generally, many factors affect tensile properties of composite materials as reinforcement types, their shape and size, interfacial adhesion between the reinforcement and the matrix, the processing methods and the processing conditions (Tezara, *et al.* 2016, 2159). It is known that E-glass fibers are hydrophilic materials because of the presence of hydroxyl groups on their surface which reduces the degree of impregnation of the fibers by the hydrophobic LDPE matrix. Poor impregnation leads to ineffective stress transfer between LDPE and E-glass fibers and decreases the mechanical properties of the produced composites. Acidic whey was used as a coupling agent to improve the tensile properties of E-glass

fibers/LDPE composites, the effects of immersion time in acidic whey on modulus and tensile strength are shown in Figure 1.

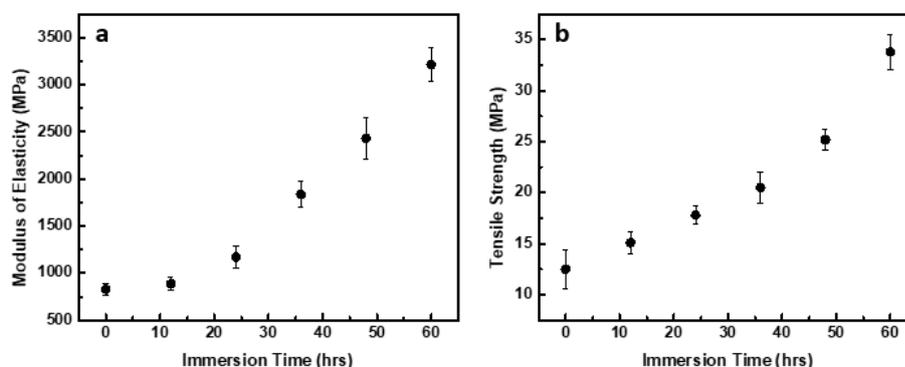


Figure (1): The effects of immersion time in acidic whey on modulus of elasticity (a) and tensile strength (b) of produced composites.

An increase in elastic modulus and tensile strength of prepared samples with increasing time of immersion in acidic whey can be observed. The use of E-glass fibers immersed for 60 hours in acidic whey increased the modulus of elasticity and the tensile strength from 827.0 ± 56.4 MPa and 20.0 ± 0.9 MPa to 3214.1 ± 176.0 MPa and 33.8 ± 1.7 MPa, respectively. The enhancement in the tensile properties is due to the amelioration in interfacial adhesion between LDPE and the fibers which improves the ability to transfer stress from matrix to reinforcement.

It was found that increasing the PE-g-MA content from 1 to 5 mass percent increased the elastic modulus of the HDPE-hemp fibers composites by 14, 50, 104, and 121 percent higher than neat HDPE, while increasing the hemp fibers content from 10 to 50 mass percent increased the elastic modulus of the composites by 23, 42, 99, and 125 percent higher than neat HDPE. The addition of hemp, on the other hand, resulted in tensile strength values of 10, 6, 31, and 48 percent lower than the neat polymer, but increasing PE-g-MA content resulted in strength enhancements of 11, 33, 61, and 71 percent when compared to uncompatibilized composites with the same amount of hemp fibers

(Roumeli, *et al.* 2015, p. 93). 8 mass% of sodium hydroxide alkaline treatment followed by silane treatment of hemp fibers improved the tensile strength and the modulus of elasticity of polyurethane-hemp fibers composites by 39% and 23%, respectively. The betterment in tensile properties was attributed to the increase in the interfacial adhesion of the fibers-polyurethane composite from 1.26 MPa for untreated fibers up to 3.18 MPa for the alkali fibers and 5.16 MPa for silane fibers (Sair, *et al.* 2017, p. 550).

It can be observed from Table 1 that E-glass fibers content in the composite materials is not constant, so it is better to observe the effect of immersion time in acidic whey on the modulus (K_E) and strength efficiency factors (K_S). The modulus efficiency factor (K_E) and the strength efficiency factor (K_S) for random distributed fibers-based composites can be calculated by modified-rule of the mixture as shown in equations 1 and 2, respectively

$$E_c = K_E E_f v_f + E_m (1 - v_f) \quad (1)$$

$$\sigma_c = K_s \sigma_f v_f + \sigma_m (1 - v_f) \quad (2)$$

Where E_c , E_f , E_m , σ_c , σ_f , σ_m and v_f represent modulus of elasticity of composite material, modulus of elasticity of fibers, modulus of elasticity of the matrix, tensile strength of composite material, tensile strength of fibers, tensile strength of the matrix and fibers volume fraction, respectively. The modulus and strength efficiency factors depend on several factors such as the fibers aspect ratio (l/d), the interfacial adhesion between the fiber and the matrix, the fiber orientation, the structure of the fibers and the void content in the composite material" (Capela, Oliveira, Pestana & Ferreira, 2017, 539).

The fibers volume fraction (v_f) can be calculated by using equation 3:

$$v_f = \frac{m_f / \rho_f}{m_f / \rho_f + m_m / \rho_m} \quad (3)$$

Where m_f , m_m , ρ_f and ρ_m are mass of the glass fibers, mass of LDPE, density of glass fibers (2540 kg/m^3) and density of LDPE (920 kg/m^3), respectively.

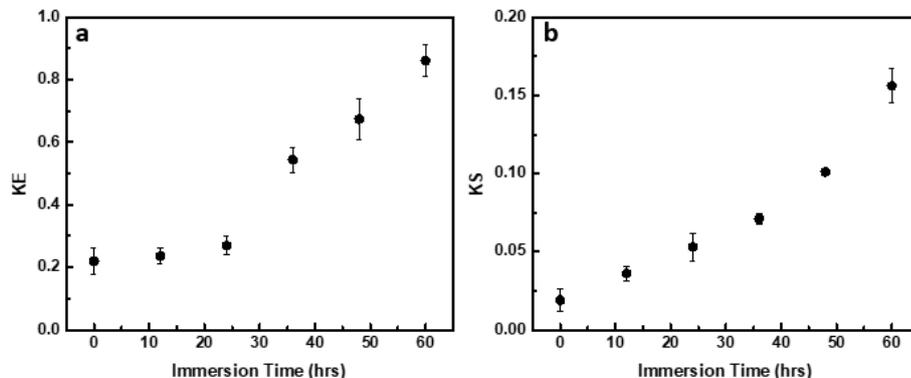


Figure (2): The effects of immersion time in acidic whey on (a) the modulus efficiency factor (K_E) and (b) the strength efficiency factor (K_S).

It is clear from Figure 2 that increasing the immersion time of E-glass fibers in acidic whey increases both the modulus (K_E) and the strength (K_S) efficiency factors of the produced composites, indicating an effective stress transfer between LDPE and E-glass fibers.

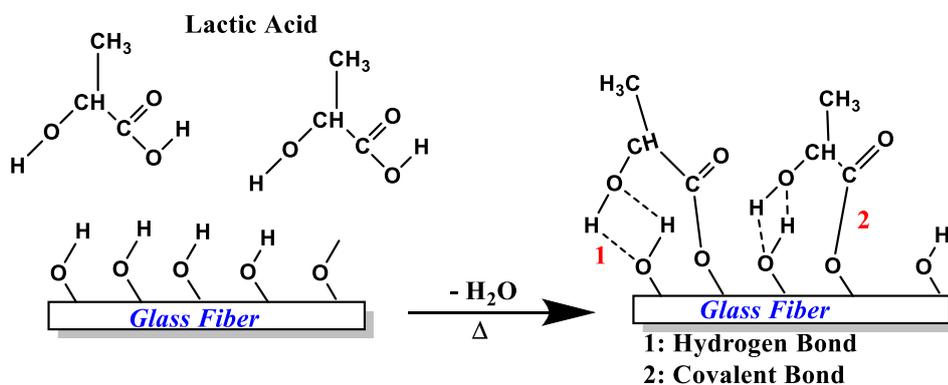


Figure (3): The chemical interactions of lactic acid with glass fiber.

Acidic whey contains lactic acid (2-hydroxypropanoic acid) which results from the fermentation of lactose by streptococcus lactis (Dosuky, Nasr, Yousef, & Barakat, 2019, p. 793). During the immersion time the presence of acidic group (COOH) on lactic acid results in a strong chemical interaction with hydroxyl groups (OH⁻) on the surface of the fibers as shown in Figure 3, while the other side of lactic acid, methyl group (CH₃) which is a hydrophobic chemical group, adheres with LDPE, this enhances the interfacial adhesion bonding between the fibers and LDPE.

On the other hand, acidic whey contains significant amount of fatty acids, casein, organic nitrogenous, collagen, starch compounds, glutamine, lysine, calcium ions and other minerals (Whitaker, 1975, p. 27). During the hot processing (200°C), fatty acids, collagen, starch compounds and the organic nitrogenous compounds hydrolyzed and form a colloidal protein and a gelatin form compound (Sanders, 2001). Casein, which is a milk-derived protein, can also form adhesive substance at high temperatures, which enhances the adhesion between the polymeric matrix and the fibers (Peppler & Perlmane, 1979, p. 100).

Also, enzymes such as trans-glutaminase (T-Gase) are involved in forming intra-molecular and inter-molecular cross-links in whey proteins, resulting in complex polymers. The resultant polymers are branched polymers that exhibit theological and functional characteristics that are different from the original whey. The polymeric whey protein has a higher active area in the interface of the fiber glass surface which results in more effective treatment increased with increasing the time (Davis & Foegeding, 2006, p. 404). The most noticeable impact of whey protein polymerization is an increase in gel strength up to 10 times than of non-polymerized whey (Whitaker, 1979, 18).

It can be seen from Figure 4 that the initial pH of acidic whey was 3.41 and then it decreases to 1.81 after 60 hours, the decrease in pH may be related to the fact that acidic whey contains bacteria and other microorganisms. The activation of these microorganisms increases the fermentation rate rapidly with increasing the immersion time in acidic

they which results in a decrease of the value of pH due to the formation of lactic acid (Virtanen, Pihlanto, Mäkinen, & Korhonen 2007, p. 106).

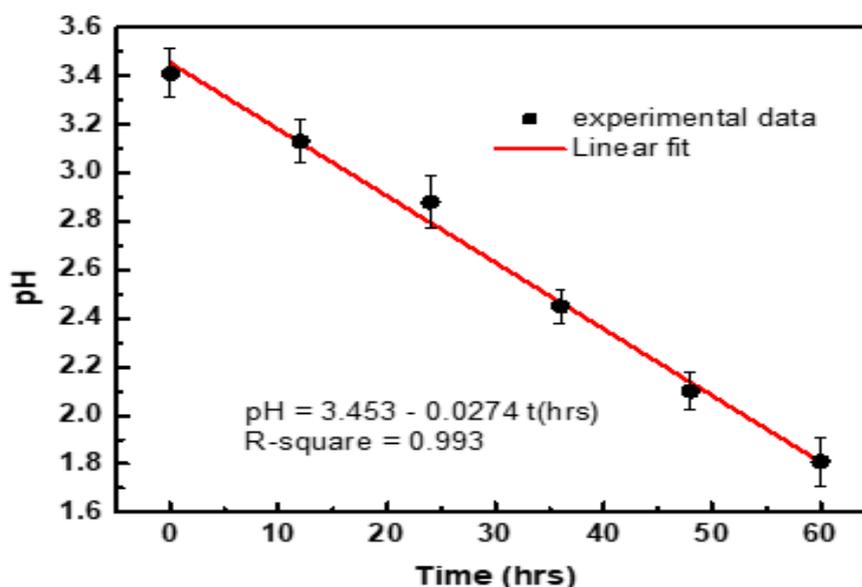


Figure (4): The effect of immersion time of E-glass fiber in acidic whey on pH.

The increment in the concentration of hydrogen ion (H^+), due to the formation of lactic acid, with increasing the immersion time of the fibers in acidic whey increases the interactions between the acidic group (COOH) of lactic acid and the hydroxyl groups (OH^-) on the surface of glass fibers as seen in Figure 3. The interactions of these chemical groups improve the interfacial-adhesion between fibers and matrix in the composite material which enhances both the modulus of elasticity and the tensile strength of the produced composites and their efficiency factors as seen from Figures 1 and 2, respectively.

Ductility Test

The ductility as percent elongation (%EL) was calculated and illustrated in Figure 5. It shows that the ductility of LDPE decreases with

increasing the immersion time of E-glass fibers in acidic whey. The brittle nature of fibers limits the plasticity behavior of composites as compared to the polymer matrix, resulting in a loss in ductility. The hydrogen bonding between the lactic acid and hydroxyl groups on the surface of glass fibers is enhanced by increasing the concentration of lactic acid with increasing the immersion time of the fibers in acidic whey. This leads to an increase in the interfacial adhesion between the fibers and the matrix and, as a result, a decrease in the ductility of the produced composites.

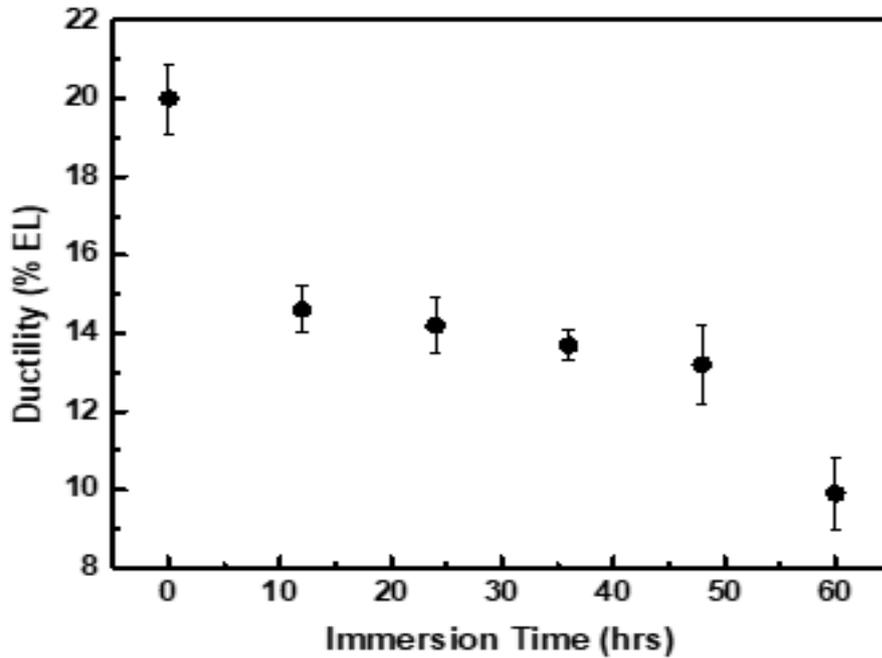


Figure (5): Effect of immersion time on the ductility of the produced composites.

The used acidic whey shows outstanding enhancements on mechanical properties such as tensile strength and elastic modulus which could be considered the highest values among other traditional or known commercial coupling agents as illustrated in Table 2.

Table (2): Comparison between acidic whey as coupling agent and other commercial ones.

No.	Polymer/Fiber Composite	Coupling Agent	Mechanical Properties	Enhancement (%)	Ref.
1	Polypropylene / glass fiber	In-situ <u>metallocenic polymerization</u>	Interfacial shear strength	210	(<u>Etcheverry & Barabosa, 2012, 1084</u>)
2	Low density polyethylene/ glass fiber	<u>Vinyltriethoxysilane (VTES)</u>	Tensile strength	109*	(<u>Fabris, Cardozo, Maular & Nachtigal 2009, 872</u>)
3	High-Density-Polyethylene/ Aramid Fiber+ Wood Flour	Dopamine and functional <u>silane VTES</u>	Tensile strength Elastic Modulus	29 10	(<u>Liu et al., 2021, 236</u>)
4	Epoxy / Palm petiole nanofiber	Amino propyl <u>triethoxysilane silanesilane</u>	Tensile strength Elastic Modulus	28* 20*	(<u>Kumar, Chandrasekaran, Santhanam & Sudharasan, 2017, p.1</u>)
5	High Density Polyethylene (HDPE)/ Acrylonitrile-butadiene rubber (NBR)/ Palm Pressed Fiber (PPF)	γ - <u>aminopropyltriethoxy silane (γ-APS)</u>	Tensile strength	29	(<u>Norizan, Santiago & Ismail, 2017, p.1</u>)
6	Low density polyethylene/ glass fiber	<u>Organofunctional silane/ maleated copolymer PE-g-MA</u>	Tensile strength Elastic Modulus	49/69 51/175	(<u>Bikiaris et al., 2001, p. 2877</u>)
7	Polypropylene-short glass fibers	PP-g-MA	Tensile strength	18	(<u>Lin et al, 2015, p. 8279</u>)
8	basalt fiber-nylon based composite	3- <u>glycidoxypropyltrimethoxysilane</u>	Tensile strength	42	(<u>Deák, Czigány, Tamás & Németh, 2010, p.590</u>)
9	Low density polyethylene/E-glass fiber	Acidic whey	Tensile strength Elastic Modulus	107 289	This work

*Values are calculated by authors because they are not reported directly.

Conclusions

It can be concluded that acidic whey can act as a novel effective coupling agent for E-glass fibers-LDPE based composites. It was observed that the use of acidic whey as a coupling agent improves the modulus of elasticity and the tensile strength of E- fibers-LDPE composites and their efficiency factors. The degree of improvement in the tensile properties and the efficiency factors depends strongly on the immersion time of E-glass fibers in whey, this was attributed to the improvement in the interfacial adhesion between the fibers and the polymeric matrix.

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