

Analysis of the Torque Required for Mixing LDPE with Natural Fibre and DCP

A. E. Delgado, W. Aperador

Abstract—This study evaluated the incidence of concentrated natural fibre, as well as the effects of adding a crosslinking agent on the torque when those components are mixed with low density polyethylene (LDPE). The natural fibre has a particle size of between 0.8-1.2mm and a moisture content of 0.17%. An internal mixer was used to measure the torque required to mix the polymer with the fibre. The effect of the fibre content and crosslinking agent on the torque was also determined. A change was observed in the morphology of the mixes using SEM differential scanning microscopy.

Keywords—WPC, DCP, LDPE, natural fibre, torque.

I. INTRODUCTION

DIFFERENT sources of natural fibre have been used in the search for new wood-plastic composites (WPCs), with their advantages over the usual inorganic fillers: availability, renewability, biodegradability, better flexibility, and easier processing [1].

Low density polyethylene (LDPE) is a “commodity type” plastic: low cost, readily obtainable, and easily processed, among other things. It has a fusion temperature of between 110 and 135°C. Its low glass transition (T_g) temperature is associated with a good retention of mechanical properties including flexibility and resistance to impact [2].

To ensure homogeneous integration of the fibre and polymer, we required the force of a motor which moved the blades of the mixer. This force or torque is proportional to the increasing viscosity of the mixture as more fibre is added to it [3]. Another factor contributing to the increase of the torque during the mixing process is crosslinking. This phenomenon occurs when a peroxide is added to the polymer. When working with thermoplastic polymers such as LDPE, it is necessary to crosslink the polymer to give it dimensional and thermic stability [4].

Through laboratory internal mixer, can be simulated process like compounding, mixing, masticating (for rubbers) that are relevant for production and processing of compounds and properties of the final product. Among the process variables, the compound viscosity, being reflected in a rotor torque, is the main physical characteristic not only with respect to the mixing process.

The viscosity is related to polymers type (thermoplastic, elastomer), reinforcing fillers, softeners, plasticizers,

processing aids, as well as to changes of mixing conditions, i.e. speed of mixer blades, temperature and pressure. Torque data can be interpreted in terms of work and energy.

The torque vs. time curve shows the characteristics of the mixing processes. The region for the torque ramping up to the peak value indicates introduction of polymers and ingredients into the mixer and start of the mixing process, torque increase due to reinforcing action of filler and because polymers that are not melt [5].

After reaching the maximum, the torque decreases due to viscosity drop caused by shear action, breaking molecular entanglements, and temperature increase. However containing compounds crosslink agents, the value of torque can again be increased by the crosslinking reaction at the end of the mixing time. This can serve as a warning to know the time and the temperature at which crosslinking agents (peroxides, sulphur, accelerants) must be added [5].

The area covered by the torque curves above the x-axis was considered the measurement of energy consumption in mixing process. It is generally desirable for the period between the curve minimum and maximum to be well expressed and to last as long as possible in order to enable thorough breakdown of filler agglomerates and as uniform as possible incorporation of the resulting aggregates into polymer matrix..

Remember that to properly set the temperature and mixing time should take into account important variables such as the geometry of the blades, rotor speed, bulk of the mixture, not least is the order of addition of ingredients the formula. You can do simulations of high shear viscosity of the extrusion process (compression and metering zones) keeping the speed of the rotors.

The objective of this paper is the study the variation in the torque when the percentage of natural fibre is increased during the mixing process as well as the crosslinking effect of LDPE by adding dicumyl peroxide (DCP).

II. EXPERIMENTAL PROCEDURES

A. Materials

Natural fibre with a particle size of 0.8-1.2mm and moisture content of 0.17%. Polymer matrix: LDPE, Polifén® 641 supplied by Ecopetrol® with MFI 1.7-2.3 g/10 min. Reticulation agent: Dicumyl Peroxide Luperox® DCP with 98% purity. In this study stearic acid was used to disperse the natural fibre within the polymer [6].

B. Formulations

Six (6) formulations were carried out in order to analyse the effects of the natural fibre content (0, 10, and 50%) and the

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addition of the crosslinking agent DCP (0, 1%), as seen in Table I.

TABLE I UNITS FOR MAGNETIC PROPERTIES						
Material	Without DCP			With DCP		
LDPE	100	90	50	100	90	50
Natural Fibre	0	10	50	0	10	50
DCP	0	0	0	1	1	1
Processing Aid	1	1	1	1	1	1
Processing Aid	1	1	1	1	1	1

C. Method

The internal mixer used for this study is composed of two 49 mm long grooved triangular rollers, 9.5 mm apart and 33.6 mm from the sides (Fig. 1). The mixer is activated and controlled by LABVIEW 8.0 software. The heating system is an element controlled by a thermocouple. It has a maximum torque of 10 N/m and a maximum capacity of 60 cm³.



Fig. 1 Internal mixer

The operating temperature range was 130 ± 5 °C, enough to soften the LDPE. Each mixture had a weight of 45 g, according to the capacity of the mixer. The mixing procedure was the following: Firstly, 50% of the LDPE was added slowly. When the LDP had melted and homogenised, we started the slow addition of natural fibre, up to 20% of the total, and after 2 min we continued slowly adding the rest of the LDPE which homogenised. We then finished adding the remaining fibre and then added the stearic acid and mixed for 10 min more. For the group of mixtures that contained DCP, this was added 1 min before finishing the mixing process. In total the time which each mixture took to finish was approximately 20 to 25 min.

The morphological analysis of the samples was carried out using differential scanning microscopy (SEM). The images were taken with a NeosCope JCM 5000 SEM microscope, the metallisation was done in a Balzers® SDC 050 sputter in pre-vacuum conditions, in the presence of argon gas in an ionising atmosphere over a gold-palladium plate. The acceleration voltage was 30 kV.

III. RESULTS AND DISCUSSION

A. Torque

Fig. 2 shows that the value of the torque is not constant across the whole mixing time, the maximum torque value is 4.8 N/m and corresponds to the mixture which has a greater fibre content (50%). The maximum torque values are seen between minutes 8 and 15 of the mixing process. The fibre content proportionally affects the torque due the elastic character of the cellulose. The increase in the torque during the first minutes is due to the incorporation of LDPE, which is introduced to the mixing chamber at room temperature and raises the mixture's viscosity due to the fact that it is in a melted state. It is slowly heated to soften it and for this reason the torque starts the diminish after 15 min, when it has incorporated all the polymer in the formulation.

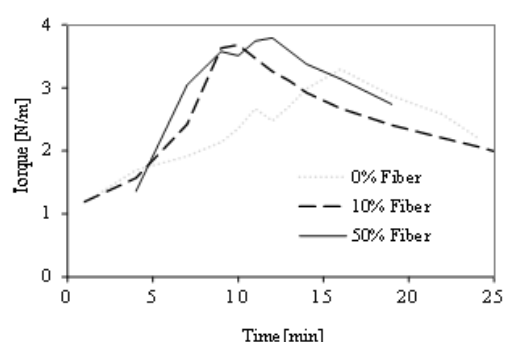


Fig. 2 Torque depending of the fibre content without DCP

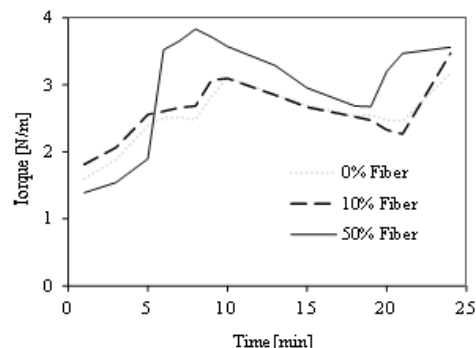


Fig. 3 Torque depending of the fibre content with DCP

Fig. 3 illustrates the incidence of the incorporation of DCP on the mixture and the effect of crosslinking. As expected, during the first stages of the process the mixture behaved in the same way as it did without the crosslinking agent (as observed in Fig. 2). However, when the DCP was added the graph shows an increase of 25% in just 15% of the time, in other words an increase in the variation of the torque which makes it possible to quantify the limiting effect on mobility of the polymer's macromolecules. The increased torque after 20 min is due to the start of the crosslinking reaction of the LDPE polymer chains. The crosslinking of the polymer diminishes the mobility of the macromolecules.

B. Morphology

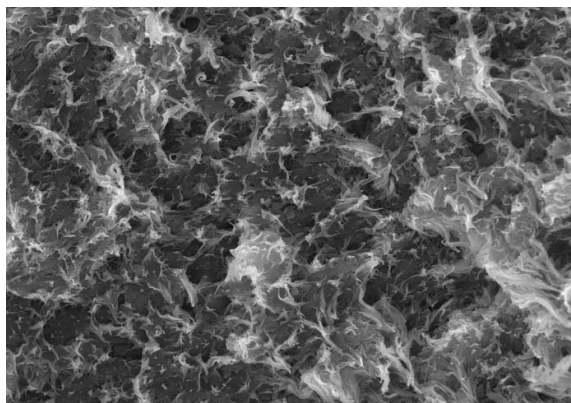


Fig. 4 SEM 1500X. Compounds after the mixing cycle, 0% fibre without DCP

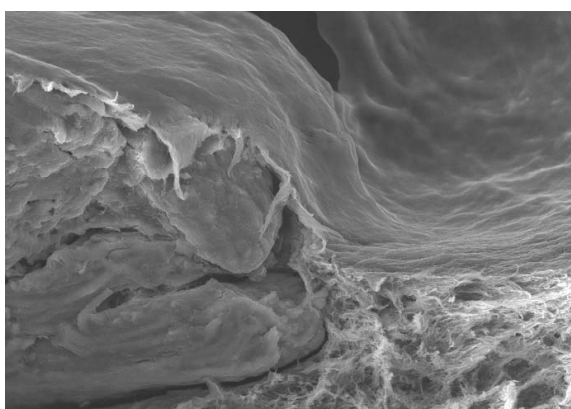


Fig. 5 SEM 1500X. Compounds after the mixing cycle, 10% fibre without DCP

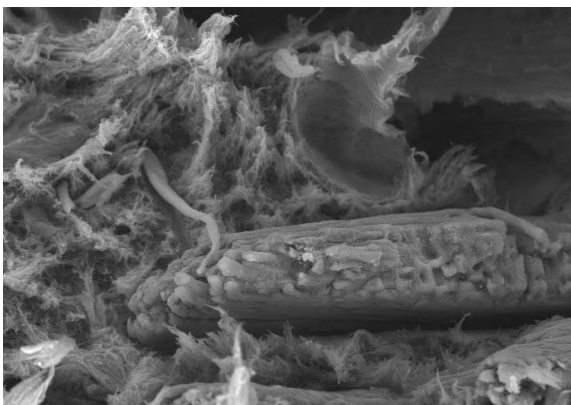


Fig. 6 SEM 1500X. Compounds after the mixing cycle, 50% fibre without DCP

A sample was taken from each formulation once the mixing cycle had ended. Longitudinal stress was applied and cross section was put under an SEM microscope.

In the samples that do not contain DCP (Figs. 4-6) the presence of filaments is observed, indicating that there was

molecular movement during the application of the longitudinal stress.

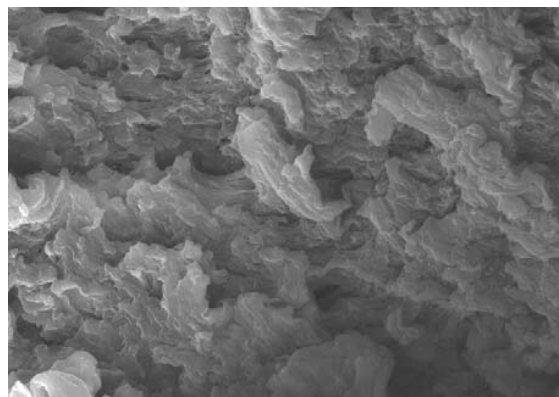


Fig. 7 SEM 1500X. Compounds after the mixing cycle, 0% fibre with DCP

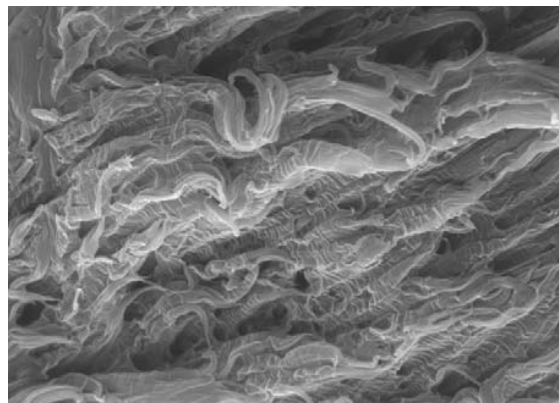


Fig. 8 SEM 1500X. Compounds after the mixing cycle, 10% fibre with DCP

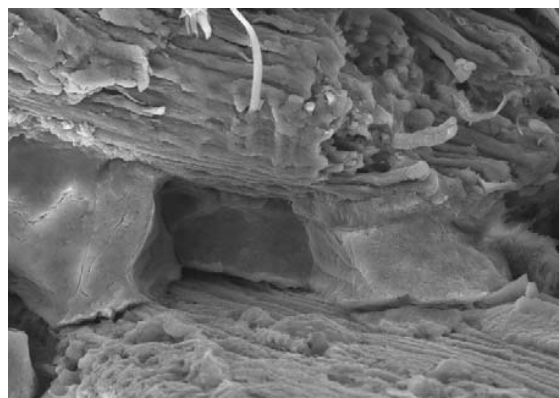


Fig. 9 SEM 1500X. Compounds after the mixing cycle, 50% fibre with DCP

In the mixtures that contain DCP (Figs. 7-9), the morphology shows a homogeneous surface with restricted molecular movement due to the start of crosslinking.

IV. CONCLUSION

The increase in the concentration directly affects the torque value. The maximum value is obtained with 50% natural fibre during minutes 8 to 15 of the mixing process, with a value of 3.8 N/m. Furthermore, when DCP is added, this also directly affects the torque value after minute 20 of the mixing process, due to the start of crosslinking. In the SEM images of the compounds which do not contain DCP, a fibrillar morphology is observed, indicating orientation or movement of the polymer chains, unlike the mixtures which contain the crosslinking agent where a homogeneous surface is observed.

REFERENCES

- [1] Xie, Y., C. Hill, Z. Xiao, H. Militz and C. Mai; "Silane Coupling Agents Used for Natural Fiber/Polymer Composites: A Review," *Composites Part A*, 41, 2010, pp. 806–819.
- [2] Martínez, N. and M. Vásquez; "Obtención y Caracterización de un Material Polimérico a Partir de la Mezcla de Polietileno de Baja Densidad (PEBD) y Almidón de Maíz Modificado," *Ing. Thesis*, Universidad Veracruzana, México, 2009.
- [3] Li, T.Q. and M.P. Wolcott; "Rheology of Wood Plastics Melt. Part 1. Capillary Rheometry of HDPE Filled with Maple," *Polym. Eng. Sci.*, 45, 2005, pp. 549 - 559.
- [4] Saiz, C.; "Fabricación de Materiales Celulares Mejorados Basados en Poliolefinas. Relación Procesado-Composición-Estructura-Propiedades," *PhD Thesis*, Universidad de Valladolid, España, 2012.
- [5] Cheremisinoff, N. and M. Dekker; "Polymer Mixing and Extrusion Technology," *J. Polym. Sci., Part C: Polym. Lett.*, 27, 1989, pp. 282-283.
- [6] Torres, F.G. and M.L. Cubillas; "Study of the Interfacial Properties of Natural Fibre Reinforced Polyethylene," *Polym. Test.*, 24, 2005, pp. 694–698.