"MICROWAVE HEATING OF POLYMERS: INFLUENCE OF CARBON NANOTUBES DISPERSION ON THE MICROWAVE SUSCEPTOR EFFECTIVENESS"

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ABSTRACT

Carbon nanotubes dispersion within the polymer matrix is a very important factor to take into account when developing new nanocomposites with optimized properties. In this article, dispersion studies have been carried out with polypropylene filled with 1% of multiwall carbon nanotubes. The nanocomposites were obtained by melt compounding in a co-rotative twin screw extruder. Processing parameters as screw speed, screw configuration and feeding technology were modified to analyse their effect onto carbon nanotubes dispersion. Developed nanocomposites were exposed to microwave heating (5.8 GHz, 700W, 60 min) and heating temperature was monitored. The relation between dispersion level of carbon nanotubes and heating effectiveness was studied. Electrical conductivity of nanocomposites was measured and used as indirect variable of microwave heating susceptor of carbon nanotubes nanocomposites.

1. INTRODUCTION

Microwaves are a part of the continuous electromagnetic spectrum that extends from low-frequency alternating currents to cosmic rays [1]. Microwaves propagate through empty space at the light speed and usually the frequency ranges swap from 300 MHz to 300GHz.At first, the microwaves were applied in navigation and radar-target detection during the Second World War. Currently the microwaves are used in a lot of industrial and different applications. The frequencies commonly used for these purposes are 915 MHz, 2.45, 5.8 and 24.124 GHz, avoiding any interference with microwave radar and telecommunications band range.

Microwave technology provides an alternative to conventional heating methodologies suitable for some widely home applications, cooking, chemical synthesis, treatment of gas effluents, curing of thermoset and rubber materials, adhesives [2], and ovens for ceramic components manufacture [3]. Basically provides an alternative methodology to conventional heating, with several advantages like: penetrating radiation, controllable electric field distribution, rapid heating, selective heating of materials, and self-limiting reactions. Two major effects are responsible for the heating which results from this interaction: dipolar polarization and conduction [4], [5]. Microwave heating present important disadvantages, mainly related to the lack of uniform and selective heating over a large volume and the transparency of most of the materials to microwaves. The lack of heating homogeneity can be reduced using alternative methodologies such as variable frequency microwave ovens (VFM) [2]. One of the most important problems detected in the microwave ovens is the presence of hot spots. Conventional fixed-frequency microwave heating suffers local fluctuations in the electromagnetic field that causes non-uniform power distribution within microwave cavities, thus leading to uneven heating and potentially poor product quality [6]. The power distribution in VFM technologies becomes more uniform because of the superposition of hundred or thousand of individual microwave modes in a few milliseconds.

The microwaves do not interact with the majority of polymeric materials due the lack of dipolar moment. For this reason, additives as heating susceptors can be used in order to prepare materials able to absorb microwaves. These additives are conductive, or have dielectric properties significantly different from the polymeric matrix. Depending on the nature of the susceptor, the material answer to the microwaves can be due to one specific mechanism or combination of several of them [7].

Different types of microwave susceptors have been reported in literature taking into account the size, shape, concentration, electrical resistivity, the distribution and dispersion of the nanoparticles in the polymeric matrix. The most commonly used susceptors are carbon structures, in particular carbon black, carbon fibers or carbon nanotubes. Other suitable particles as silicium carbide, titanium dioxide, metal flakes, zinc oxide or talc have been tried [8]. Harper et al. [9] studied the influence of carbon black as microwave testing particles with different surface area. They concluded that morphology characteristics of carbon black had a strong influence on their microwave susceptor properties. The heating efficiency of carbon black particles increases when the surface area of the particle

is higher. The work reported by Qincai Ling et al. [10] was focused on the microwave effect of short carbon fibers dispersed in polyethylene blends. H.C.Shim et al. [11] and R.Xie et al. [12] worked on the development of carbon nanotubes coatings on polymeric substrates. In these papers, the microwave radiation increases the temperature of the polymer surface due to the microwave susceptor effect of carbon nanotubes. Temperature is increased until the polymer is melted which helps to form the carbon nanotube coating. The effect of microwave on carbon nanotubes has led to a controversial discussion between different authors. A. Wadhawan et al. [13] proposed a mechanism where the residual metals (Fe) from the synthesis of carbon nanotubes are responsible of their microwave susceptor effect. However, other works has shown the independence of microwave effect on carbon nanotubes from residual metals [14].

The aim of the work was to study the effect of carbon nanotubes as dispersed microwave susceptors in a polymeric matrix. Polypropylene was chosen as matrix due its non-polar nature and microwave transparency and the nanocomposites were prepared by melt mixing procedure using a co-rotative twin screw extruder. The effectiveness of heating process was tested taking into account the nanotubes dispersion in the polymer matrix Microwave technology can be a high energy efficiency fabrication method for producing plastic products as it will be demonstrated in this study.

2. EXPERIMENTAL

2.1. Materials

Commercial multiwall carbon nanotubes (MWCNT)NC7000 were purchased from Nanocyl. These MWCNT are produced via catalytic carbon vapor deposition (CVD) process with an average diameter of 9.5 nm, length of 1.5 μm and carbon purity around 90%. Homopolymer polypropylene was selected as polymer matrix. The selected grade was PP DUCOR 1101S from DUCOR Petrochemicals. This material has a MFR (230ºC/2.16 kg) of 25 g/10min, a tensile modulus of 1500 MPa and a melting point of 163ºC.

2.2. Sample preparation by melt compounding

Nanocomposites were obtained in a co-rotative twin screw extruder COPERION W&P ZSK25. This extruder has a diameter of 25 mm and an L/D ratio of 40. Nanocomposites with different dispersion grade were obtained by varying different processing parameters. Two different screw configurations were tested in order to study the influence of the shear stress in the dispersion of MWCNT: highly dispersive or high shear (HS) and highly distributive or low shear (LS).High shear screw was designed employing large number of kneading blocks with wide disks and high angle between disks. Low shear screw was achieved with low number of kneading blocks. Moreover, two processing routes were evaluated: direct feeding of MWCNT powder (direct compounding) and feeding MWCNT in masterbatch form (masterbatch dilution). Finally, four different values of screw speed: 100, 300, 600 and 800 rpm were tested. The concentration of carbon nanotubes in all nanocomposites was 1%w/w. The temperature setting from hopper to die was set with the following profile for all the nanocomposites: 260ºC/220ºC/220ºC/210ºC/200ºC/190ºC.The different factors and variables applied in this study are summarized in Table 1

Table 1: Parameters studied during compounding process

Factor	Variable
Screw configuration	Highly dispersive (HS)
	Highly distributive (LS)
Screw speed	100 rpm
	300 rpm
	600 rpm
	800 rpm
Processing route	Direct compounding (DC)
	Masterbatch dilution (MB)

A masterbatch with a MWCNT loading of 15% was produced under the following optimized conditions: highly dispersive screw configuration, 600 rpm and temperature profile 230 – 190 ºC and subsequently used in the masterbatch dilution method

Samples with dimensions of (10 x 1 x 0.4) cm were obtained by compression moulding in a hot press (COLLIN model P200E) at 200 ºC/15 bars during 15 min. These samples were used in morphology characterization and electrical conductivity studies.

2.3. Characterization techniques

The quality dispersion of MWCNT in the nanocomposites was examined by optical microscope. Samples with disc shape of 2.5 cm of diameter and 100µm of thickness were prepared in a hot plate press at 210ºC for 3 minutes. The microscope employed was LEICA model DMRX equipped with software of image analysis (Leica Materials Workstation V 3.6.3). The parameters measured in the samples were: mean particle size and agglomerates density.

Scanning Electron Microscopy (SEM) studies were performed with a Phenom Pro X desktop microscope. The microscope works at multiple acceleration voltage (5, 10, 15 kV), reaching a resolution of less than 12 nm. The samples were prepared by cryogenic fracture of compression moulding test bars and subsequent coated with gold by sputter coating technology.

TEM measurements were performed using a high-resolution transmission electron microscope (JEOL JEM-1010) equipped with software of image analysis MegaView III. Samples were analyzed at a voltage of 100 KV and 79mA..Compression moulding samples were cut with an ultramicrotome Reichert & Jung FC4E (Reichert Microscope Services, USA) and the nanocomposite ultra-thin sections of about 100nm were place on a200-mesh copper grid.

Electrical conductivity of compression moulding test bars was measured using the two-probe method a multimeter KETHLEY 2000 according to the procedure described in the standard UNE-EN ISO 3915.Silver electrodes were painted on the samples in order to improve contact between the sample and measuring electrodes.

2.4. Microwave heating

The microwave heating trials were carried out in a 5.8 GHz multimode microwave provided from the German company FRICKE&MALLAH. The maximum power of the microwave was 700W and the oven cavity had the following dimensions: (300x320x195) mm. Samples in pellet form were exposed to microwaves for a period of 1 min at a maximum power. This process was repeated six times for each sample and five temperature data were obtained each time. Mean reached temperature was calculated with all the values obtained.. All the samples were placed in the center of the cavity to ensure heating homogeneity, which was assessed with a thermochromic disc paper. Temperature increase in the microwave chamber was monitored with an infrared camera FILR ThermaCAM P640 (ε=0.95).

3. RESULTS AND DISCUSSIONS

3.1. Analysis of the MWCNT dispersion for different processing conditions

Dispersion of carbon nanotubes in the nanocomposites was analyzed with an optical microscope as a first approach of morphology studies. At optical microscope level, the number of agglomerates can be compared between different samples. Carbon nanotubes tend to agglomerate during melt compounding and these agglomerates are bigger when the quality of the dispersion decreases. It's well known that the most influencing factors to obtain small agglomerates sizes in polymer composites loaded with nanofillers are screw speed and compounding methodology.

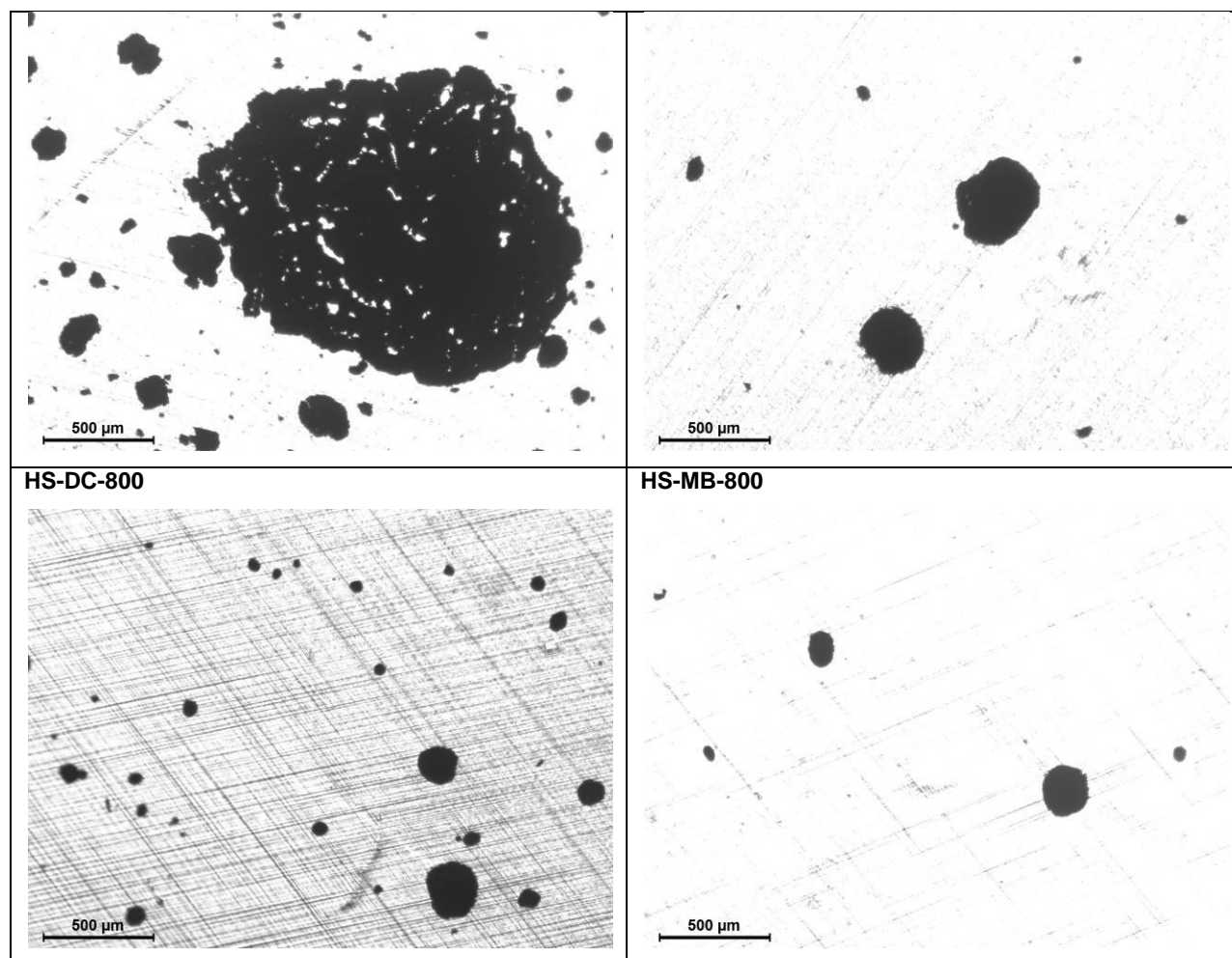| LS-DC-100	LS-MB-100

HS-DC-800 **HS-MB-800**

Figure 1: Optical microscope images x25: Low shear screw configuration-direct compounding-100 rpm (up left), low shear screw configuration-masterbatch-100 rpm (up right), high shear screw configuration-direct compounding-800 rpm (down left), and high shear screw configuration-masterbatch-800 rpm (down right)

In order to compare the processing routes using in this work: masterbatch technology (MB) and direct compounding (DC), pictures of samples obtained at different shear rates are showed in Figure 1. As it can be seen in pictures, the masterbatch technology improves dispersion even at low shear screw configuration and low screw speed.

Big agglomerates of MWCNT's with sizes higher than 500 µm can be seen when nanocomposites are processed with a low shear screw configuration and low screw speed by direct compounding. Lower agglomerates size is achieved at high screw speeds. Agglomerates size reduction is more pronounced in direct compounding technology than in masterbatch technology. The agglomerates size is reduced when screw speed is increased as a consequence of a high shear stress. It can be said that long residence time and high shear stress decreases agglomerates size and improves dispersion, being the residence time the most critical factor for achieving a good dispersion. Masterbatch dilution increases the residence time of carbon nanotubes inside the extruder as the marterbatch is processed two times. This fact make less critical the processing conditions as it is seen in Figure 2, where similar agglomerates size is achieved at high and low shear screw configurations.

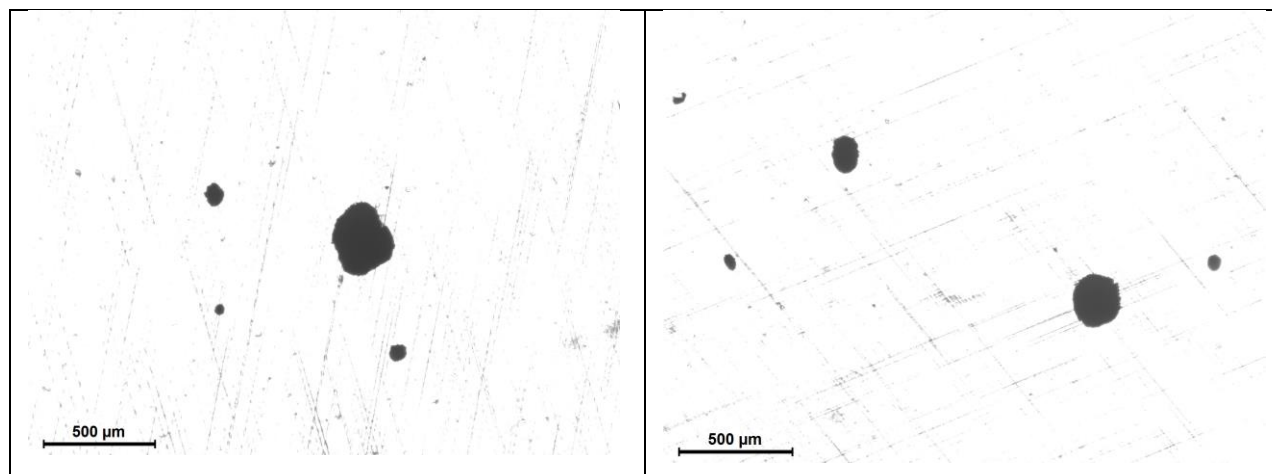| LS-MB-800	*HS-MB-800*

Figure 2: Optical microscope images x25: Low shear screw configuration-masterbatch-800 rpm (left), high shear screw configuration-masterbatch-800 rpm (right)

Mean particle size and agglomerates density from optical microscope images were analyzed in order to evaluate the MWCNT dispersion. These data have been represented in Figure 3 and Figure 4. It is shown in Figure 3 that for all nanocomposites the mean particle size decreases as the screw speed increases. Moreover, attending to the two processing routes employed (methodologies) the particle size reduction is more effective with masterbatch technology (MB) in comparison to direct compounding (DC). Incorporation of carbon nanotubes into a marterbatch implies that nanofillers have been exposed to two processing or cycles of extrusion. Thus, the residence time of carbon nanotubes inside the extruder is longer and they are exposed to shear forces for longer time. As a result, the screw configuration and the screw speed are less influencing factors on the final mean particle size in the compound. Nevertheless, feeding carbon nanotubes in powder form makes the nanocomposite more sensible to shear stress inside the extruder. Figure 3 shows that the mean particle size of compounds produced via direct compounding are optimized in terms of reducing the mean particle size by increasing screw speed and employing a highly dispersive screw (HS) configuration.
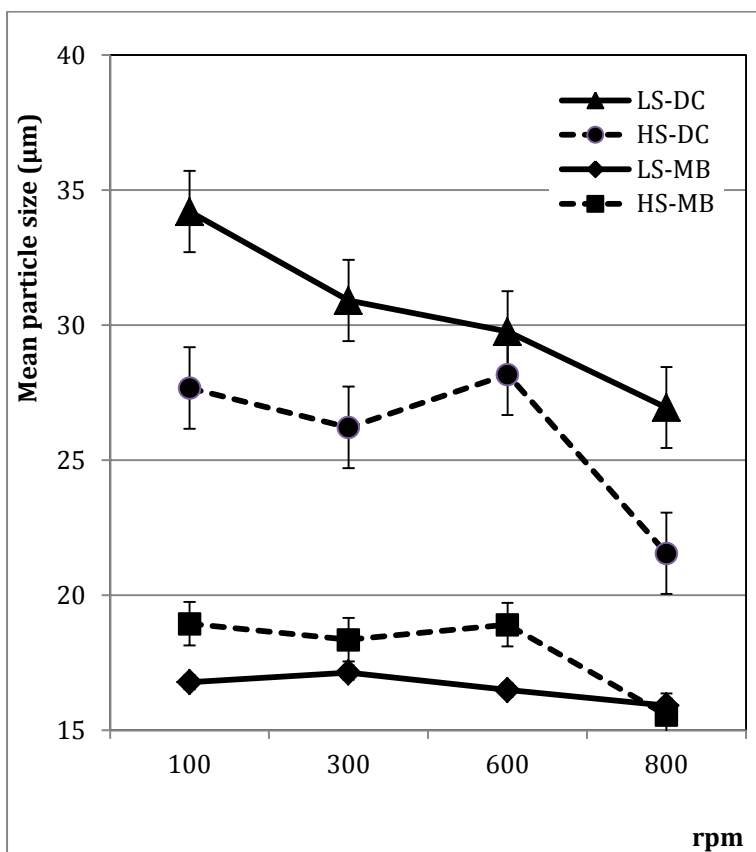
Figure 3: Mean particle size versus screw speed for different screw configurations and methodologies of processing (similar processing temperature)

Agglomerate density makes reference to the area of the sample, which is occupied by aggregates in relation to the total area of the sample. It is calculated with the following formula [18]:

$$Aglomerates\ density\ (\%) = \frac{A_x}{A_0} \times 100 \qquad \text{Eq. 1}$$

Where A_x is the area of the sample with CNT agglomerates and A_0 is the total area of the sample.

The agglomerate density is a good indicator of the dispersion of agglomerates into the polymer matrix. Figure 4 shows the influence of processing conditions on MWCNT agglomerate density. As happened with the mean particle size, masterbatch technology is the most influencing factor for decreasing agglomerates density. In fact, there is no representative difference between high shear and low shear screw configurations even when varying the screw speed of the extruder. It is very important to point out that when a good masterbatch of carbon nanotubes is employed, the processing conditions during dilution to obtain the final nanocomposite have lower effect on the carbon nanotubes dispersion. The opposite situation takes place when working with direct feeding of carbon nanotubes in powder shape (direct compounding DC). In this case, a MWCNT agglomerates uniform dispersion is obtained at high screw speed and employing a highly dispersive screw configuration. In this way it is possible to decrease agglomerate density to a lower level close to that obtained with masterbatch technology.
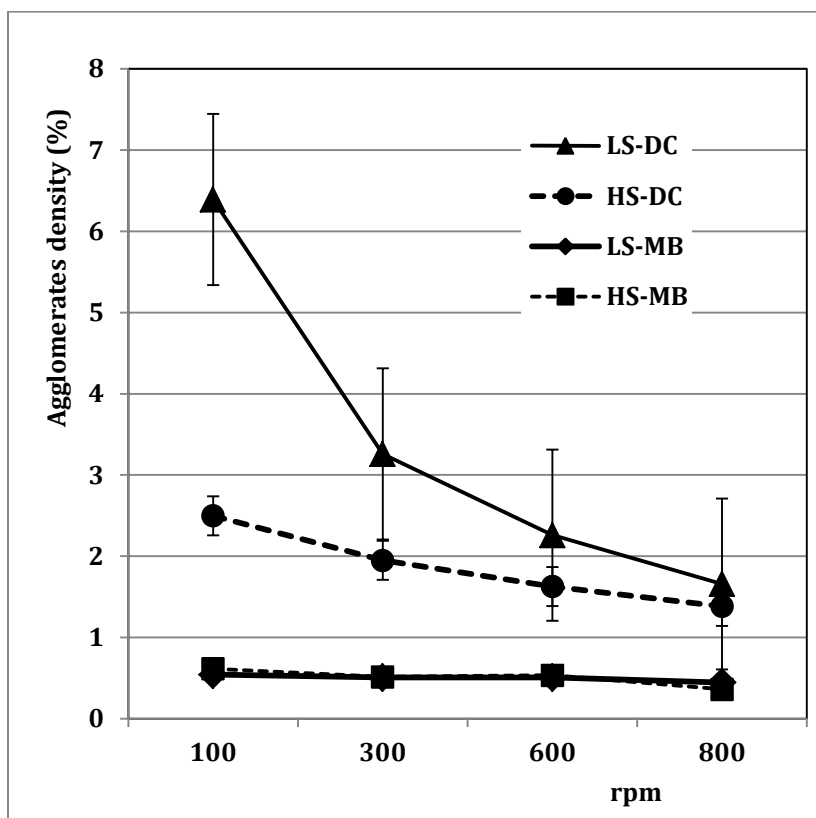
Figure 4: Agglomerates density size representation versus screw speed for different screw configurations and processing methodologies

Another method for comparing processing conditions with carbon nanotubes dispersion is to analyze the specific mechanical energy. The specific mechanical energy of a compounding process makes reference to the level of shear stress that has been applied to the material during the compound production. It is expressed in kWh/kg as a value of the energy applied per kilogram of compound in one hour, Equation2. It is a very important factor as it allows the comparison between the shear stress levels of different process. Specific mechanical energy (SME) was calculated for each process and the values obtained were correlated to the agglomerates density.

$$SME = \frac{kW \times T\,(\%) \times \frac{rpm}{max\,rpm}}{Q(\frac{kg}{h})} \times \mathcal{E}(\%) \quad \text{Eq. 2.}$$

Where kW makes reference to the extruder power engine, T is the torque observed during extrusion (referred to the maximum torque of the extruder), rpm is the screw speed, Q makes reference to the production rate and ɛ is an efficiency factor of the engine (usually 97%)

High specific mechanical energy is needed to decrease agglomerates density as it is shown in Figure 5. A higher specific mechanical energy is related with a lower the agglomerates size in the nanocomposite. When the nanocomposites are produced via direct compounding, as residence time of carbon nanotubes is low, high shear levels are needed to compensate this effect. Nevertheless, the dispersion of carbon nanotubes produced with masterbatch is better as agglomerates density is lower even at low SME values. Therefore, the dispersion of carbon nanotubes is less sensible to shear stress when increasing the residence time inside the extruder by working with masterbatch technology.
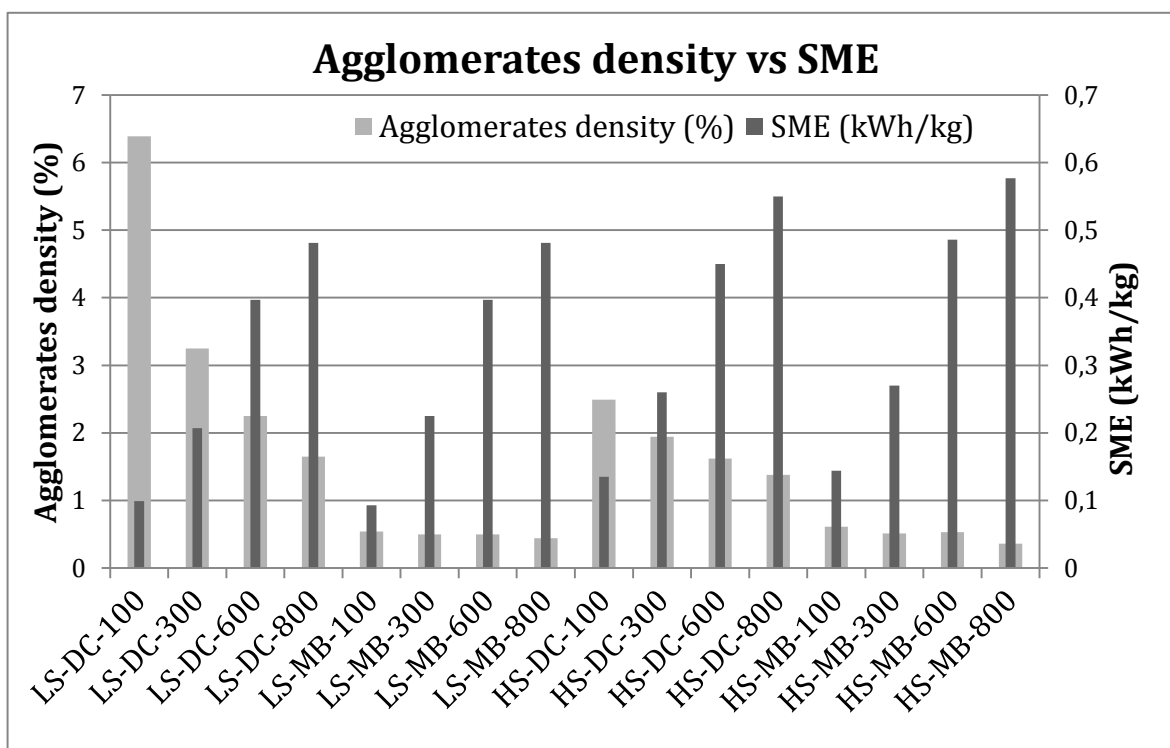
Figure 5: Agglomerates density versus specific mechanical energy

Scanning electron microscope analysis was carried out to analyze the influence of residence time in the agglomerate size. Figure 6 shows the micrographs at different magnifications of the nanocomposites produced by direct compounding and a nanocomposite produced via masterbatch. Both nanocomposites were produced employing a high shear screw configuration and the extrusion was carried out at 800 rpm. Therefore, the difference between both samples is the time that the polymer matrix and carbon nanotubes have been exposed to shear stress. It can be seen that high residence time decreases the carbon nanotube agglomerates size. This effect is seen in Figure 6a and 6b where there is less carbon nanotubes agglomerates present in the sample compared to direct compounding. High magnification pictures are very helpful to analyze the size and morphology of the agglomerates. At high magnification, direct compounding picture shows a big agglomerate of carbon nanotubes. All the area of the picture is a big agglomerate. Nevertheless, in Figure 7a and 7b the agglomerates are smaller even at higher magnification.
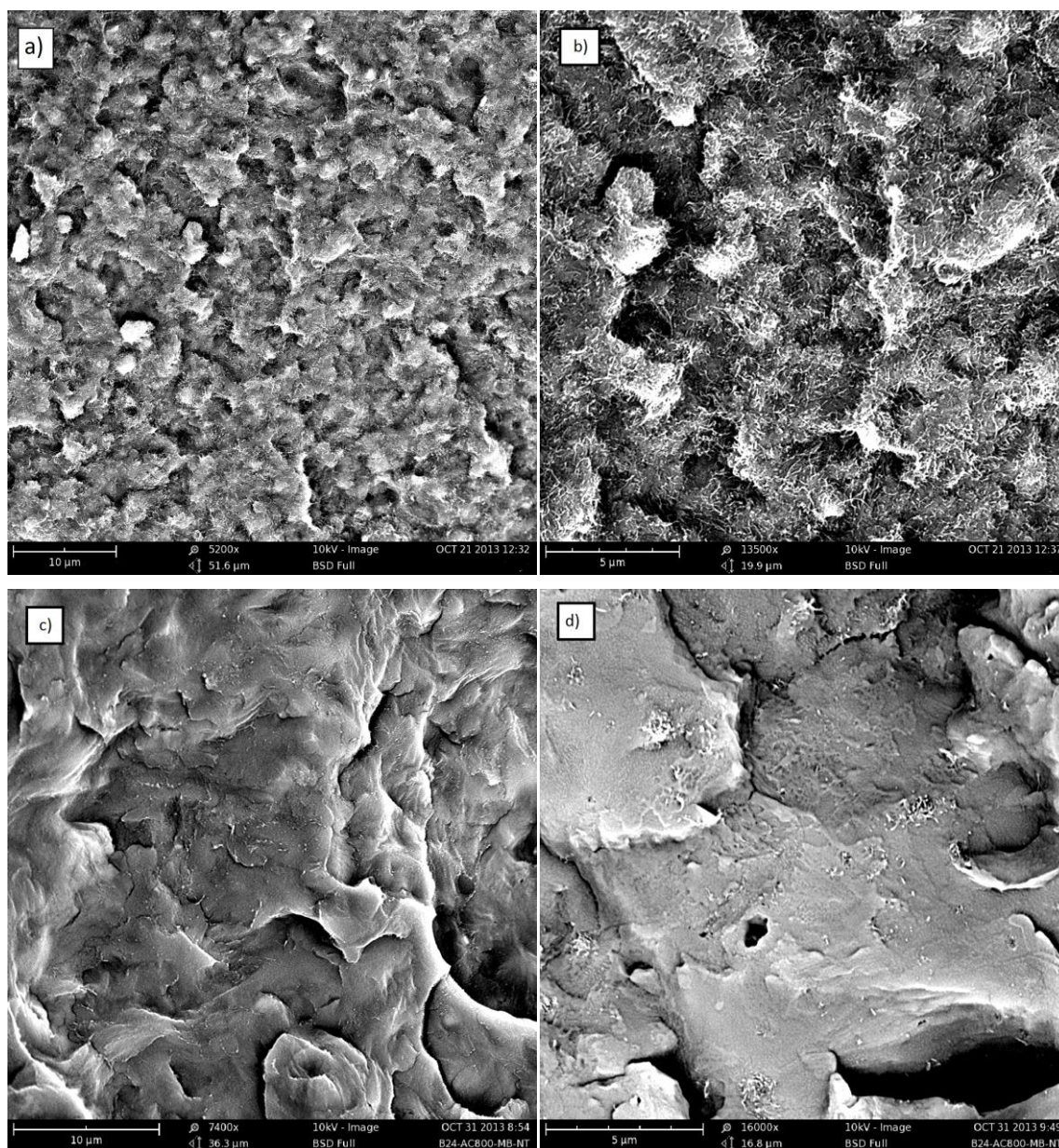
Figure 6: SEM micrograph of nanocomposite samples: (a) HS-DC-800 Low magnification, 5200x; (b) HS-DC-800 High magnification, 13500x; . (c) HS-MB-800 Low magnification, 7400x; (d) HS-MB-800 High magnification,16000x.

Transmission electron microscopy was used to assess MWCNT length and dispersion in the nanocomposites. Masterbatch technology improves dispersion of MWCNT but it was important to evaluate how longer time exposed to shear stress has an influence on the length of carbon nanotubes[19]. Figure 7 shows MWCNT in the nanocomposite produced by direct compounding and via masterbatch. At high magnifications (x10000) long carbon nanotubes can be seen in the TEM image when the nanocomposite is produced with direct compounding. Nevertheless, the nanocomposite obtained via masterbatch has shorter carbon nanotube length. Long residence time of MWCNT inside the extruder and high screw speed of sample HS-MB-800 are too high which may cause the breakdown of MWCNT. Even though MWCNT may be broken at high shear and residence time, these processing conditions improve the dispersion of MWCNT within the polymer matrix which may lead to a better electrical properties of the nanocomposite and better microwave susceptor properties.
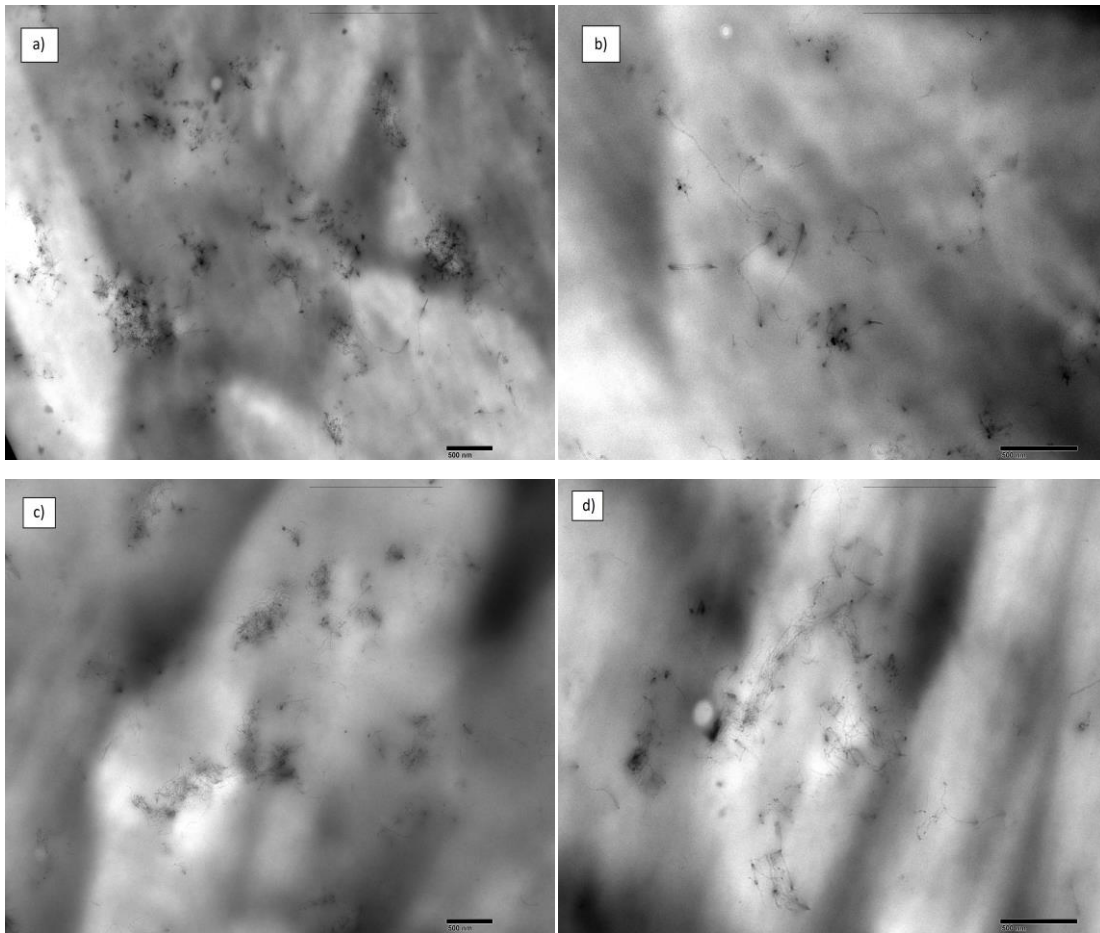
Figure 7: TEM micrograph of nanocomposite samples: a) HS-DC-800, 6000x; b) HS-DC-800, 10000x; c) HS-MB-800 , 6000x; d) HS-MB-800, 10000x

Big agglomerates also reduce the effectiveness of carbon nanotubes as they actuate as microparticles loosing the advantages of incorporating nanoparticles.

3.2. Effect of processing conditions on electrical conductivity

Figure 8 shows the surface electrical conductivity of nanocomposites obtained at different processing conditions. Nanocomposites with better dispersion have better electrical properties than those compounds in which MWCNT are forming bundles. In general, better properties are achieved when nanotubes are feeding in masterbatch form. Electrical conductivity is less affected by shear stress inside the extruder. It can be observed that nanocomposites processed at different shear conditions can achieve the same electrical properties when the residence time is increased. Therefore, when working via masterbatch there would be no reason for applying high screw speeds as there is no extra benefit on the electrical properties and carbon nanotube length is reduced, as was confirmed by TEM analysis. The effect of MWCNT breakdown is not shown in electrical properties as the improvement of MWCNT dispersion compensates the reduced length of MWCNT.
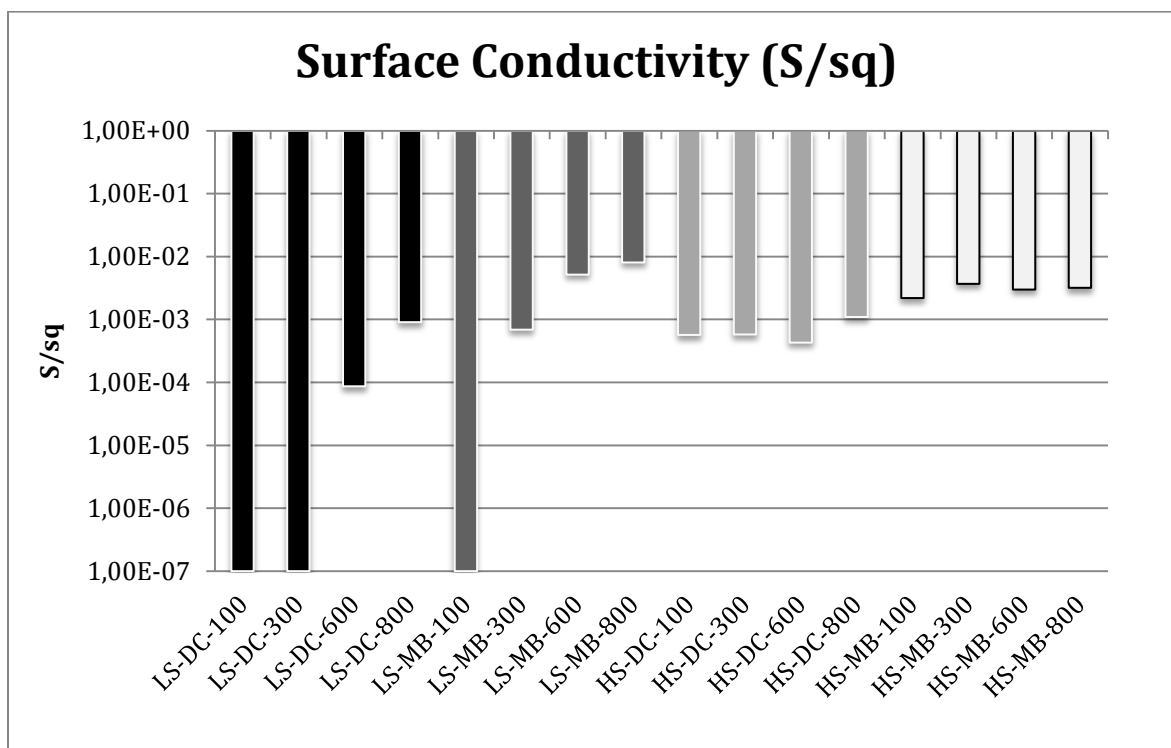
Figure 8: Surface electrical conductivity of nanocomposites produced at different processing conditions

3.3. Effect of processing conditions on microwave heating temperature

Nanocomposites with different dispersion qualities were exposed to microwave heating at 700W for 60s. After this time, the mean temperature was analyzed.. Maximum and minimum temperatures were also compiled to evaluate the heating homogeneity. Figure 10 illustrates the heating temperature reached by each nanocomposite.It is well known that it is difficult to achieve an homogeneous heating with microwaves. It this case high values of the maximum temperature in comparison with the mean temperature demonstrates the presence of a hot spot. In Figure 9 it can be seen that nanocomposites produced via masterbatch exhibit approximately the same temperature regardless the screw speed applied during processing. Meanwhile, the nanocomposites produce via direct compounding show an increase in the final temperature as the screw speed increases. The highest temperature value belongs to the samples produced at low shear and direct compounding. This could be related with the worst dispersion that can produce hot spots in the sample during microwave heating.
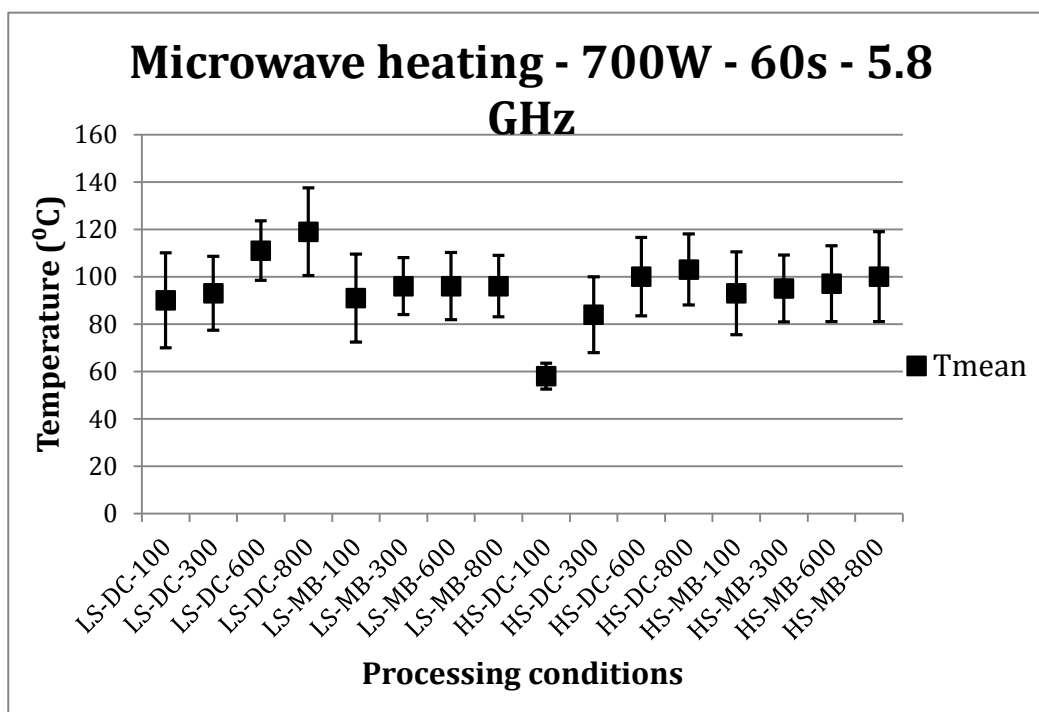
Figure 9: Nanocomposites temperature after 60s of microwave heating at 700W (5.8GHz)

In order to study the influence of dispersion on the microwave heating effectiveness, the mean particle size and agglomerate density of the nanocomposites have been represented versus the mean reached temperature. Figure 10 and 11, show the relation between the mean particle size and the agglomerate density for different processing conditions.
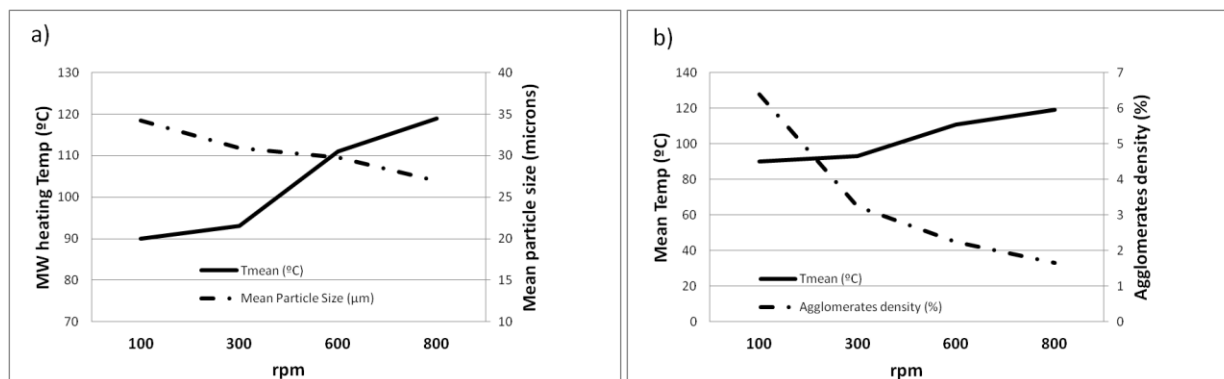

Figure 10: Microwave heating temperature versus mean particle size (a) and agglomerates density (b) for nanocomposites processed by Low Shear-Direct compounding (LS-DC) at different screw speeds

As can be appreciated in Figure 10, the microwave heating temperature increases as the mean particle size and agglomerate density of MWCNT in the nanocomposites decreases. Lower particle size and agglomerate density implies a better dispersion of MWCNT. Improving dispersion of MWCNT the electrical conductivity of the nanocomposite increases as well as its behaviour as microwave susceptor.

Thus, a higher extrusion speed is related to the increase of the heating effectiveness of carbon nanotubes as microwave susceptors. The same behaviour is observed in nanocomposites processed at different conditions (Figure

9,10 and 11). Nonetheless, it has to be pointed out that when increasing shear stress and residence time (high shear screw configuration and masterbatch feeding technology) the heating homogenity increases. This effect can be seen in Figure 13 where the difference between the temperature at 100 rpm and 800 rpm is less important than in those nanocomposites processed at low shear and via direct compounding (Figure 10).
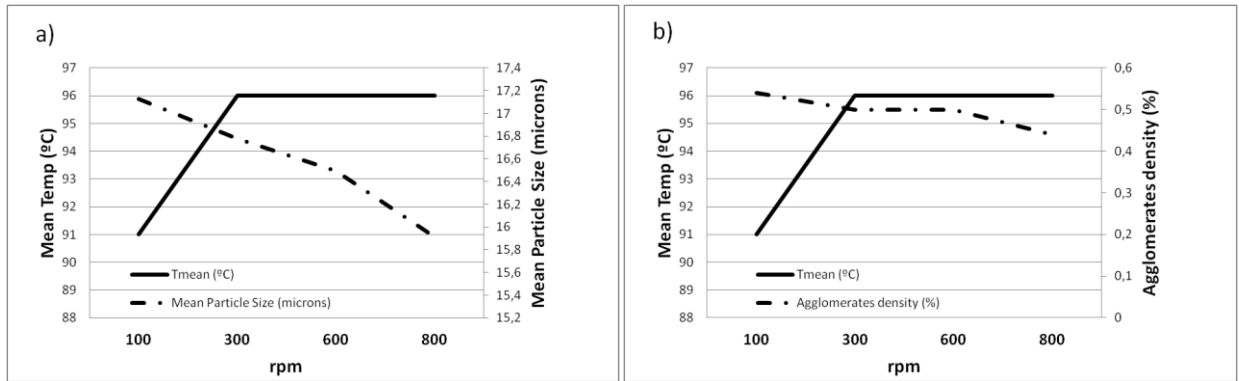

Figure 11: Microwave heating temperature versus mean particle size (a) and agglomerate density (b) for nanocomposites processed by Low Shear Masterbatch (LS-MB)at different screw speeds
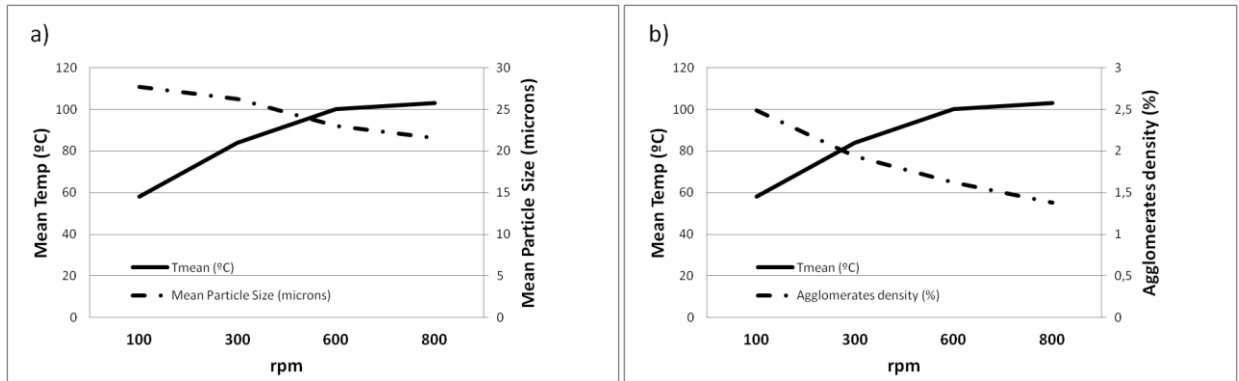

Figure 12: Microwave heating temperature versus mean particle size (a) and agglomerate density (b) for nanocomposites processed by High Shear Direct Compounding (HS-DC) at different screw speeds

Figure 13 shows the susceptor behaviour of the nanocomposites obtained via masterbatch technology where the best CNTs dispersion was achieved. In this Figure, the reached temperature in the microwave increases as the particle size and agglomerate density decreases, and on the other hand, the lower susceptor particle size increases the heating effectiveness.
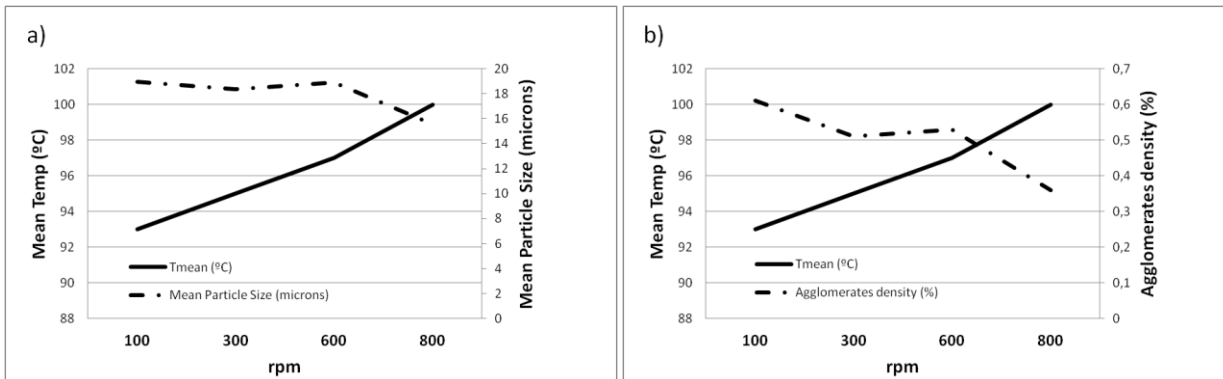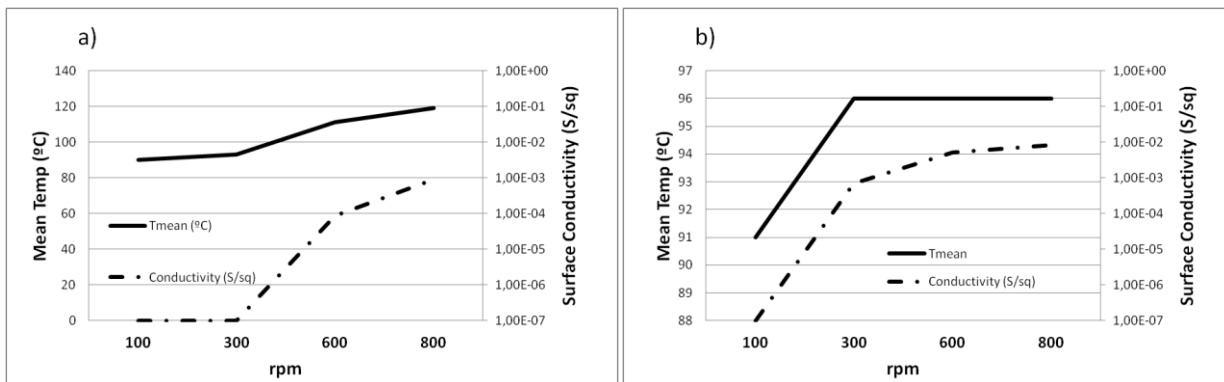
Figure 13: Microwave heating temperature versus mean particle size (a) and agglomerate density (b) for nanocomposites processed by High Shear Masterbatch (HS-MB) at different screw speeds

3.4. Electrical conductivity versus microwave heating temperature

Surface electrical conductivity of the nanocomposites can be correlated with microwave (MW) susceptor effectiveness of the nanocomposites. In figure 14 it can be seen that electrical conductivity is a suitable indirect measurement of the heating capacity of nanocomposites when exposed to microwaves. The heating effectiveness increases as electrical conductivity is higher. An increase in the reached nanocomposite temperature in the microwave is correlated to a higher electrical conductivity of the nanocomposite.

A significant increase of the electrical conductivity is achieved with increasing screw speed, as well as heating temperature. Using high shear screw configurations and high residence time working by masterbatch route it reduces the effect of screw speed onto the electrical conductivity. Therefore, a better dispersion of carbon nanotubes is translated to a higher electrical conductivity and a higher microwave susceptor effectiveness.
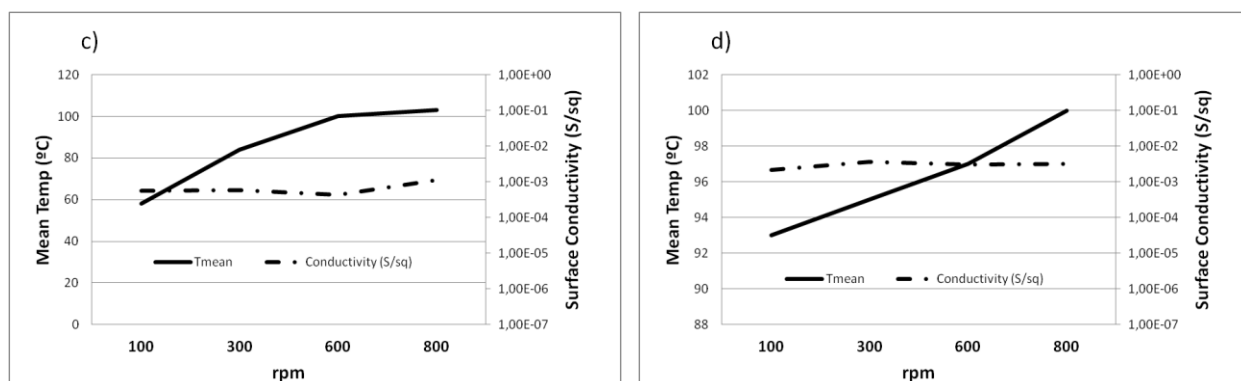
Figure 14: MW heating temperature versus surface conductivity at different processing conditions: (a) Low shear-direct compounding, (b) Low shear-masterbatch, (c) High shear-direct compounding, (d) High shear-masterbatch.

4. CONCLUSIONS

Polypropylene with 1% of carbon nanotubes were produced via melt compounding with different processing conditions. Screw speed, screw configuration and feeding methodology were highly influencing factors on the dispersion level of carbon nanotubes. Dispersion level was analysed measuring mean particle size and agglomerate density.

The dispersion achieved was more homogeneous when the nanocomposites were produced by masterbatch route, since the residence time of carbon nanotubes inside the extruder is more high, and the influence of screw speed and screw configuration on dispersion of carbon nanotubes is reduced. Therefore, low screw speeds and distributive screw configurations can be employed for nanocomposites production. In case of direct feeding of carbon nanotubes, the dispersion is improved by increasing screw speed, which can be set as the most influencing factor in this case.

Degree of dispersion of carbon nanotubes had a big influence on the electrical conductivity. Higher electrical conductivity values were obtained when the nanocomposites were processed via masterbatch technology at high screw speeds (600 rpm and 800 rpm). Electrical conductivity was related to the microwave susceptor effectiveness of the nanocomposites. In this sense, higher temperatures were achieved in the microwave with those nanocomposites that had better electrical conductivity. Therefore, electrical conductivity of nanocomposites was evaluated as indirect measurement of microwave heating effectiveness. Thereby, higher heating temperatures were obtained as mean particle size and agglomerate density decreased.

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