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Cork Plastic Composite Optimization for 3D Printing Applications

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Abstract

Among natural fillers, cork has been acknowledged as a suitable alternative of other cellular materials that are widely employed in engineering applications due to their low conductivity to heat, noise and vibration, high abrasion resistance and flexibility, high compressibility ratio, among other characteristics [1]. The eco-friendly features of natural fillers based composites make them a very promising and sustainable solution to large markets mainly if additive manufacturing technologies, such as 3D printing, are used [2]. Through 3D printers, engineers, designers and architects can create design and decor products with a free complexity of geometry. In this research work, plastic matrices of HDPE – obtained from conventional suppliers – were reinforced with different ratios of cork waste and natural cork powders – obtained from cork transformation industries – to find the optimum mixture for 3D printing. The effects of cork powders content in the plastic on the morphological, physical and mechanical properties of the composites were investigated through the density, optical microscopy, wettability, thermal analysis and tensile testing. Cork-based composites were processed by an extrusion system, and the mixture of polymer, adhesive and fillers is discussed. The results show that the addition of pure cork and cork waste can be processed with polymers such as HDPE, having adequate physical and mechanical properties.

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Keywords: Cork plastic composite; Cork powder; High density polyethylene (HDPE); Coupling agents; Extrusion; 3D Printing

1. Introduction

The highlight given to filler-reinforced thermoplastics, in particular polymer matrices filled with natural organic fibers instead of the synthetic ones, to improve the physical and thermal properties of polymers, has been increasing considerably in the last years due to their versatility for potential applications, ranging from automotive, building, medical, aerospace and electronics industries [4-6]. Natural organic fillers are well known by their low density, environmental and raw materials low costs; they contribute for the reduction of the pollutant emission; at the same

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time, they are fully degradable not representing a health risk and also they can be assumed as a renewable resource. Among natural fillers, cork has been acknowledged as a suitable alternative of other cellular materials that are widely employed in engineering applications due to their low conductivity to heat, noise and vibration, high abrasion resistance and flexibility, high compressibility ratio, among other characteristics [1]. Portugal is responsible for 52% of cork world production, followed by Spain. Together representing around of 70% of cork worldwide production [6, 7, 8]. As a result of cork processing to manufacturing different cork products, cork industries produce a great amount of wastes, which representing 25–30% of the raw material [7]. These wastes are, in general, cork powder due to the reduced cork dimension particles. Cork powder has been used as combustion fuel, as a way for removing heavy metals from industrial waste waters, as agricultural subtract or for the preparation of activated carbons to obtain microporous materials [1, 6, 7, 8].

Currently new cork based materials – Cork Polymer Composites (CPC) – that represent a great innovation potential for engineers, architects, designers and other professionals have been developed [9, 10, 11]. CPC has been produced as a result of cork powder blended with several polymers, mainly polypropylene (PP) and polyethylene (PE), by using extrusion or pultrusion methods. CPC are, in general, further processed by injection or compression molding. Cork particle amount and dimensions, coupling agent amount and type, the relative humidity and manufacturing process among others determine the physical and mechanical properties of CPC. Several strategies can be followed in order to overcome the natural cork-polymer incompatibility problem improving their interfacial adhesion and, consequently, increasing the CPC mechanical performance [8, 9, 10, 11].

Current CPC production processes impose limitations on the complexity of the geometry and shape of the parts to be produced, which in turn greatly restricts the creativity of designers and consequently, the characteristics of differentiation and innovation presented by new products on the market. Additive Manufacturing (AM) technology allows the rapid, automated and fully flexible manufacturing of products from templates generated by CAD (Computer Aided Design) in a fast, automated and fully flexible manner. The absence of tools translates into an almost total freedom in the generation of complex geometries, which offers a versatility which is non-existent with current productive systems. This allows designers to create physical forms of immeasurable complexity from the generation and optimization of three-dimensional digital models (3D) [12-14]. Thus CPC are a very promising and sustainable solution to a large market mainly if AM technologies, such as 3D printing, are used [2, 8].

Although the global core of this work is focused on the customized CPC components, in this paper only the procedure to find optimal mixture for 3D printing is presented. Physical and mechanical characterization of the CPC produced is also discussed. Adjustable and optimal CPC printing parameters specification will be presented in a next publication.

2. Materials and methods

2.1 Materials

2.1.1 Cork

The most important byproduct of cork processing is cork powder [1, 6, 7, 8]. Due to its calorific value is used as raw material in industrial processes and its monetary value is insignificant. There are several types of cork powders: grinding powder, granulation powder or pre-grinding powder; cleaning powder, without impurities; the finishing powder from the cutting and sanding operations. In this case different types of cork powder were used. One of them was provided by Amorim Cork Composites facilities in Portugal and consists mainly in pure cork granulometry with an average particle size of 0.5-1 mm. The other one came from floor covering manufacture processing waste, a result of an industrial process and presents contamination with varnish, cork, wood fiber, polyvinyl chloride (PVC), polyurethane (PU) among others.

2.1.2 Polymers and coupling agents

The polymer used as matrix was a high density polyethylene (HDPE KS10100) with a melt flow index (MFI) of 4g/10 min-1 (190ºC; 2.16 kg) and a specific weight of 0.955 g/cm^3 provided by DOW. The coupling agent consisted in a HDPE modified with maleic anhydride (Fusabond E265) with a MFI of 12g/10 min-1 and a specific weight of 0.95g/cm^3 supplied by DUPONT.

2.2 Processing

2.2.1 Sieving

Pure cork was supplied with proper granulometries to be used in this experimental work between 0.5-1 mm and 2-3 mm. To get separation of the granules by size between 0.5-1 mm on cork waste, a mechanical sieving process was carried out, so that to proceed to the mixtures to obtain the composite materials.

2.2.2 Drying

Both the pure and cork waste powders were dried using an oven at 90ºC, in order to get similar moisture conditions on the processed materials. This procedure also ensures a better dimensional stability and improves the process since not all the cork contains the same moisture content and different moisture contents affect the final product to be developed.

2.2.3 Mixing and Twin-Extrusion compounding

The two different cork powders (pure and waste) were mixed with the polymer using different proportions (Table 1) and compounded using a extruder machine (Werner & Pfleiderer-ZSK25x38D) with a 25 mm diameter spindle, 1 main feeder, 1 side feeder, 1 degasser, a 10 kW motor and a maximum spindle speed of 300 rpm. In addition to these systems for mixing and extrusion, the machine is also constituted by a tank with rollers and water for material cooling and a system that allows cutting the material in order to obtain the CPC granules. Since cork begins to have a significant degradation behavior at temperatures higher than 200ºC [15] an extrusion temperature at 180ºC was maintained.

Table 1. Compositions of the cork polymer composites

Cork polymer composite type	HDPE (wt%)	Cork waste (wt%)	Pure cork (wt%)	Coupling agent (wt%)
HDPE	100	0	0	0
CPC1	80	0	15	5
CPC2	65	0	30	5
CPC3	80	15	0	5
CPC4	65	30	0	5
CPC5	45	50	0	5

2.2.4 Injection molding

The CPC granules were dried in the oven at 90ºC and then injected using a proper machine (Boy 22A) designed for the automatic processing of thermoplastic materials, elastomers, silicones among other thermoplastic materials to create the samples needed for the mechanical testing. The samples were injected with a mould temperature between 180ºC and 195ºC depending on the polymer, cork type, and cork percentage.

2.3 Cork powders and CPC density

The density of cork powders and CPC granules obtained after extrusion was calculated on the basis of the Archimedes principle from which a partially or totally submerged object in a liquid, is subjected to a force, in the vertical direction, from bottom to top, and with intensity equal to the weight of the liquid displaced. Given that the cork has lower density than water it was difficult to measure the density using a pycnometer. Therefore, in order to conduct the test, cork was forced to submerge in the liquid using a capsule especially build for this process. The test was conducted using pure cork with two different granulometries (0.5-1mm and 2-3mm) and cork waste (0.5-1mm). Nine specimens were tested for each cork type and five specimens were tested for each CPC granule type. The normality of the density results were evaluated using the Shapiro-Wilk test as well as an ANOVA variance analysis to compare the density between cork types and granulometry.

2.4 Optical microscopic morphology

Optical microscopy was employed to evaluate the cork particles distribution on the CPC specimens. The CPC samples were observed using an optical microscope MICROS MCX100 connected to a digital camera. The samples were prepared taking one slice of CPC material with a scalpel from the longitudinal section of the tensile specimen.

2.5 Wettability

The degree of wetting (wettability) was determined using a contact angle test measured through the contact between the liquid and the CPC solid surface. The water contact angles were measured with Tensiometer Theta Lite TL100 using a water droplet as the indictor. When measuring the contact angle of the sample a water droplet was first deposited by a syringe on the sample surface, and then the projection of the sessile droplet was captured to calculate the contact angle. Five measurements on different sample spots were made for each specimen.

2.6 Tensile Tests

The mechanical performance of the cork composites was evaluated through the tensile strength test. This test was conducted with a universal test machine Instron 4505. The machine consists of two spindles with ball bearings that allow the arm to move in the vertical direction (Z axis) and through the load and displacement sensors system in order to return values to which it is subjected to test a particular sample. The dimensions of the test pieces are in accordance with the standard ISO 527-4 [16].The test was conducted using a load cell with maximum capacity of 100 kN and a crosshead speed of 2 mm/s. The dimensions of the specimens were 4 mm thick, 150 mm length and 20 mm width. Five specimens were tested for each CPC material and all of them were loaded until the core break. The results were analyzed regarding the maximum tensile stress and strain. The elastic modulus was obtained for all the specimens using the first 5 to 80 collected stress-strain data points, given that in the initial phase of the process the grips may not be properly adjusted to the sample resulting in a deviation of the initial measurements.

2.7 Thermal Analysis

A STA 6000 (Perkin Elmer) was used for thermal analysis of the materials. Samples of 6 mg were placed in alumina pans and empty pans were used as reference. All samples were first heated at a range of 30–160 ºC at a heating rate of 10 ºC/min and held isothermally for 1 min to mitigate any prior thermal history. Afterwards, the samples were cooled to 30 ºC at 10 ºC/min and then reheated to 160 ºC at the same rate. After each test, the melting point region from the thermograph was analyzed to determine the heat of fusion (ΔHm) and the melting temperature (Tm); the crystallization region was analyzed to determine the crystallization temperature (Tc) of all samples. To evaluate the thermal degradation of the materials, the samples were exposed to a temperature ramp from 30 ºC to 600 ºC, at a heating rate of 10 ºC/min. The flow rate of nitrogen was 20 mL/min during all the runs.

3. Results and Discussion

3.1 Cork powders characterization

All the cork types studied follow a normal distribution based on the Shapiro-Wilk test results. Considering the analysis of variance (ANOVA) there was no clear evidence of the influence of granulometry on density as the samples follow a normal distribution (P>0.05). On the other hand, pure cork and cork waste powders show a significant difference in the density results (P<0.05), probably due to the presence of debris in cork waste.

Table 2. HDPE, cork and CPC materials density results

Material	Density(g/cm^3)
Pure cork (2-3 mm)	0.279 ± 0.096
Pure cork (0.5-1 mm)	0.226 ± 0.026
Cork waste (0.5-1 mm)	0.450 ± 0.113
HDPE	0.955(*)
CPC1	0.73 ± 0.230
CPC2	0.66 ± 0.130
CPC3	0.70± 0.120
CPC4	0.61 ± 0.050
CPC5	0.83 ± 0.150
(*) Density of HDPE from the polymer Data Sheet	

Density results for CPC granules (Table 2) showed a normal distribution (P>0.05) confirming that variation within the tests weren´t statistically significant, showing a standard deviation higher than the expected. These results can be due to a constraint caused by the extrusion process which produced CPC with different material distribution. However, these results suggest a decrease in the mean density with the increase of cork from 15% to 30%.

3.2 Optical microscopic morphology

It was observed that the CPC´s with pure cork presented a better distribution of the cork in the polymer matrix than those with cork waste (Fig. 1). Similar results were reported by Fernandes et al. [9]. A possible explanation for this behavior could be the density, given that pure cork density is approximately half of the cork waste density, resulting in much higher volumetric amount of pure cork in the mixture. Fig. 1c) and d), related with the CPC´s composed of cork waste, also suggest that there is a presence of other debris, but the optical microscopy images of the powders were not enough to identify the composition and distribution of the debris in the mixture.

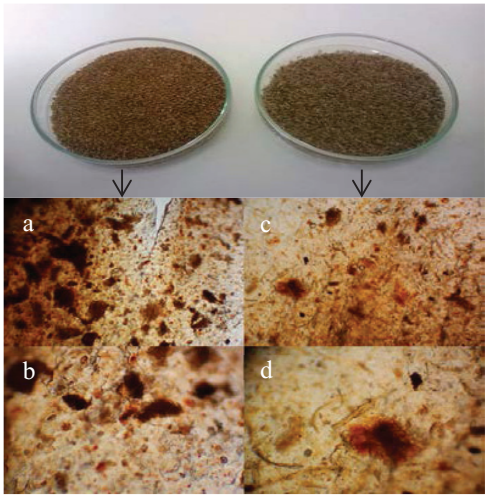

Fig. 1. Optical micrographs showing CPC (a) with pure cork with 40x magnification (b) with pure cork with 100x magnification (c) with cork waste with 40x magnification (d) with cork waste with 100x magnification

3.3 Wettability

The purpose of the contact angle assays was to study the degree of wetting when a solid and a liquid interact. Small contact angles (lower than 90º) correspond to high wettability, while large contact angles (greater than 90º) correspond to low wettability. From Fig. 2 it is observed that all samples show mean wettability lower than 90º, which is lower than what is reported in literature that indicates HDPE has a contact angle close to 90º [18]. This should be due to some porosity in the surface of the specimens after the injection process.

The CPC´s materials with the addition of pure cork (CPC1 and CPC2) showed a clear difference between them which means that with the increase of cork percentage, the contact angle begins to diminish and the fluid starts to spread over a large area on the surface. The CPC's with cork waste (CPC3, CPC4 and CPC5) show higher contact angles than the pure cork ones, which indicates that the wetting of the surface is unfavourable. So the fluid will minimize its contact with the surface and form a compact liquid droplet [17]. On the other hand, the others CPC´s, with the addition of cork waste, present very close values of contact angle making difficult its analysis. During the experiment and with a close observation of the CPC's surface it was possible to see that there was a clear difference between certain zones which could be explained by the injection processing of the samples. This could be also a possible cause for the high deviation of the contact angle results.

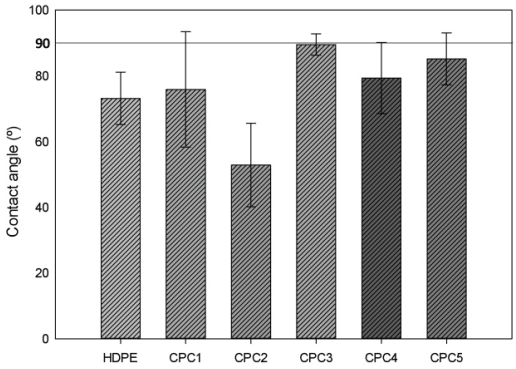

Fig. 2. Contact angle of HDPE and various CPC materials using HDPE matrix

3.4 Thermal Analysis

Table 3 summarizes the crystallization temperature (Tc), melting temperature (Tm), heat of fusion (ΔHm) of the neat polymer and composites as determined from DSC curves. The DSC cooling curves of the HDPE and CPC's are shown in Fig. 3a). The HDPE crystallization peak temperature is at 112 ºC. With the incorporation of pure cork and cork waste the crystallization temperatures present almost the same value comparing with neat polymer. Regarding Tm values of the CPC´s with the addition of cork materials a small increase of the temperatures were detected. On the other hand, the results of ΔHm provide important information about crystallinity and show a decrease of the values in the samples with concentration greater and equal to 15% (w/w) of cork powders. In conclusion, CPC's containing suberin and lignin revealed lower crystallinity, which can also be explained by the heterogeneous and only physical mixture of the materials [10].

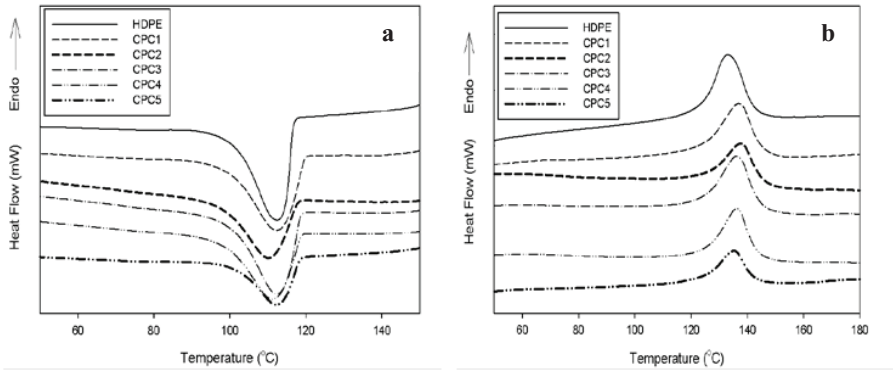

Fig. 3. Figure X DSC curves of the samples: first cooling cycle (a) and second heating cycle (b).

The TGA and DTG (first derivative of the TGA) curves of HDPE, pure cork, cork waste and CPC's under nitrogen atmosphere are presented in Fig. 4a) and 4b), respectively. Relevant data from all materials are summarized in Table 4. The chemical degradation of pure cork and cork waste starts at 270 ºC and 250 ºC, respectively. This result suggests that cork based materials can be processed with polymers having melting temperatures lower than this value, such as the HDPE. Additionally, results from Fig.4 a) show that when cork powder and cork waste are mixed with HDPE the thermal stability of the polymer decreases.

Analyzing DTG curves and Table 4 it is observed that for pure cork and cork waste, three maximum peaks were detected, for pure cork the third peak is the one with large intensity at 420 ºC (weight loss: 41%), for cork waste is

the first peak at 288 ºC (weight loss: 29%). These results corroborated with those reported by other authors [10, 19]. Regarding pure cork, the first and second peaks should correspond to the degradation of fractions of the two major cork components, lignin and suberin. The third one can be attributed to suberin, the most thermal resistant component in cork [10, 19]. The cork waste, which is composed of cork, wood and other waste polymers, shows as expected a slightly different behavior due to the presence of different species on their composition. The degradation starts earlier which is related with wood constituents, such as hemicellulose, lignin, and cellulose. Studies have shown that the degradation of wood constituents occurs between 250 and 350 ºC [20].

Comparing CPC's samples, pure cork composites (CPC1 and CPC2) DTG curves demonstrate a single decomposition temperature peak (T max1) at ≈480 ºC with a weight loss of 98% (CPC1) and 93 % (CPC2). Cork waste composites (CPC3 and CPC4) reveal two degradation peaks, the first one corresponds to cork waste and the second peak with HDPE. Increasing the concentration of cork waste to 50% (w/w) (CPC5), the DTG curve demonstrates a third peak probably due to the wood components [20].

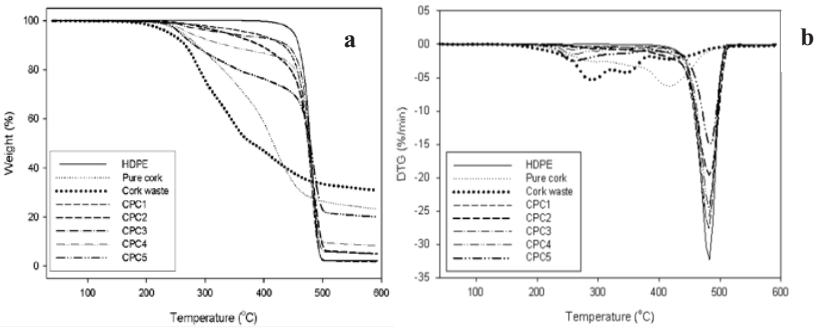

Fig. 4. (a) Thermogravimetric analysis of the samples (b) Derivate Thermogravimetric curves of the sample

Table 3. Thermal properties of the tested specimens

		HDPE	Pure cork	Cork waste	CPC1	CPC2	CPC3	CPC4	CPC5
DSC	Tc (ºC)	112,23±0,82	-	-	111,49±0,75	109,99±0,61	112,43±0,79	111,65±0,96	111,66±0,60
	Tm (ºC)	133,28±0,54	-	-	137,17±0,40	137,67±0,51	135,93±0,34	137,23±0,81	136,13±0,95
	ΔHm (j/g)	147,25±7,79	-	-	137,50±7,47	107,32±4,06	136,78±0,95	108,79±1,94	74,86±5,07
TGA	**T on 1** (ºC)	462,32±0,44	267,36±5,95	251,86±2,23	456,46±0,70	450,81±2,93	243,67±1,91	243,24±1,54	238,29±0,49
	T on 2 (ºC)	-	-	-	-	-	462,23±1,04	464,16±2,16	465,77±1,02
	Mass loss 1 (%)	99,77±0,24	16,98±1,21	28,75±5,11	97,53±0,24	93,63±1,23	6,31±0,45	11,66±0,57	14,21±0,37
	Mass loss 2 (%)	-	14,20±0,41	19,13±9,77	-	-	89,16±0,12	78,04±0,50	5,82±1,20
	Mass loss 3 (%)	-	41,14±2,16	17,51±0,66	-	-	-	-	60,65±2,74
DTG	T**max1** (ºC)	483,76±0,70	278,02±2,03	288,49±5,20	480,68±1,78	484,84±0,22	253,91±1,61	257,89±2,23	254,97±1,71
	T**max2** (ºC)	-	353,31±1,21	352,30±4,18	-	-	482,16±2,31	483,55±1,01	347,79±0,71
	T**max3** (ºC)	-	420,11±0,88	420,28±2,80	-	-	-	-	484,18±0,55

3.5 Mechanical properties

Table 4 summarizes the most important results obtained from tensile tests. The tensile strength and the maximum strain were significantly reduced in all cases comparing with the neat polymer. The decrease in tensile strength can be partially due to the incompatibility between cork particles and the polymer matrix. The adhesion between the cork powder and the matrix limit the applied load resulting in a decrease of tensile strength. The CPC´s specimens in general showed that there was evidence of a correlation between stress and strain data. With the increase of cork percentage there was an increase of stress and a decrease of strain for both the pure cork and cork waste composites. The CPC´s with cork waste showed evidence of how much the density influence the results, given that cork waste powder is twice as much heavier as pure cork powder resulting in more volumetric quantity of polymer in the mixture, what should be the reason for the higher tensile strength and elastic modulus obtained for the cork waste composite. The CPC´s with pure cork showed a decrease of elastic modulus with the increase of cork addition, although for the cork waste CPC´s it was observed a different behaviour. With the increase from 15% to 30% of cork waste there was an increase in the mean elastic modulus, but with the increase from 30% to 50% the mean elastic modulus decreased, having however higher variability.

Table 4. Tensile properties of HDPE and HDPE cork polymer composites

Materials	Strain at break(%)	Tensile Strength(MPa)	Elastic Modulus(MPa)
HDPE	-	24.30 ± 0.09	944.60 ± 72.65
CPC1	31.70 ± 2.30	16.79 ± 0.13	625.60 ± 43.60
CPC2	21.20± 1.40	17.26 ± 0.10	569.40 ± 70.86
CPC3	52.70 ± 4.30	18.62 ± 0.25	665.42 ± 34.08
CPC4	10.70 ± 1.30	19.36 ± 0.18	951.60 ± 50.61
CPC5	3.70 ± 0.70	21.00 ± 0.98	851.58 ± 120.25

4. Conclusions

One of the purposes of this work was to compare the influence of different cork powders mixed with a HDPE matrix. The results taken from the cork powders density measurements showed that cork waste density is higher than the pure cork, leading to higher amount pure cork in the composites mixture than for the composites with cork waste. A higher percentage of cork waste can therefore be added to the polymer matrix.

As for the CPC density there was some disparity of the results obtained resulting from the heterogeneous distribution of the cork on the polymer matrix. This behavior contributed to the high standard deviations on the CPC's contact angles making it difficult to understand the real influence of the cork addition. Nevertheless, the CPC´s with cork waste presented a lower wettability than the ones with pure cork.

Thermal properties showed that the addition of pure cork and cork waste can be processed with polymers such as HDPE. In terms of mechanical properties all CPC´s had a lower tensile strength than HDPE. In general, with the addition of cork all the CPC's presented an increase of tensile strength and a decrease of strain. The CPC´s with cork waste held more load than the CPC´s with pure cork.

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