Morphology, Physical, and Mechanical Properties of Particleboard Made from Rape Straw and Wood Particles Glued with Urea-Formaldehyde Resin

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The morphology, physical, and mechanical properties were investigated for single-layer particleboard made with various proportional contents of wood and rape stalk particles glued with urea-formaldehyde resin. Fine crushed rape stalk particles were used for the experimental particleboards. The weight ratios of rape-to-wood particles were 0:100, 10:90, 30:70, 50:50, and 70:30. Mixed beech and spruce wood particles with percentages of 30% for beech wood and 70% for spruce wood were considered for the configuration. Urea-formaldehyde resin with a solid content of 66 ± 1% was added to the single-mat configuration at a level of 12%, based on the weight of the particles. Physical (density, water absorption, and thickness swelling) and mechanical (modulus of elasticity, bending strength, internal bond strength) investigations on the particleboards obtained in the laboratory conditions were conducted. The results were compared to the requirements of the EN 312 (2004) standard. In addition, morphological observation at macro- and microscale and vertical density profile analysis was conducted on particleboards in order to characterize the interaction between woodrape particles and resin. Variations of the structure and density were observed relative to thickness, providing information concerning to the internal bond performance of the panels.

Keywords: Particleboard; Rape stalk; Wood particles; Physical properties; Mechanical properties

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INTRODUCTION

Rapeseed crop is gaining popularity every year among Romanian farmers, becoming a profitable crop due to relatively strong market viability and stable yields. With a planted area of approx. 600,000 hectares and an increased production reaching around 1750 tonnes, Romania is one of the main rapeseed cultivators and exporters in Europe according to GAIN Report RO1709 (2017). Oilseed rape is used for biodiesel production. At present, rape straws are used for burning in heat or electricity production, for animal bedding or composting if the plants are not chemically treated. Habashescu and Cerempei (2012) showed that two thirds of the harvested rape plants are represented by stems and leaves, and only one third by seeds. Thus, rape straw (RS) shows considerable promise as crop residue biomass.

Chemical content of rape straw stems consists of cellulose, hemicellulose, and lignin (Dziurka *et al.* 2005; Potůček and Milichovský 2011; Huang *et al.* 2016), similar to wood (Szczepkowski *et al.* 2007). This is the reason that much research is conducted

nowadays for using this renewable resource as an alternative to wood in particleboard and fiberboard manufacturing.

Particleboard production in Romania reached 2,800,000 cubic meters in 2017 (Faostat 2017), and wood demand for this sector is high, while great efforts are being made to save forests. In this context, particleboard manufacturers are interested to find alternative raw materials for replacing wood as much as possible, without affecting the technological process. Rape straw could be an alternative to wood as a raw material to produce boards.

In recent years, research on using rape straw to produce boards with synthetic resins was conducted. Dziurka *et al.* (2005) made experimental boards with 100% rape straw particles using four types of binding agents, namely urea-formaldehyde (UF), phenol-formaldehyde (PF), melamine-phenol-formaldehyde (MPF), and diphenyl diisocyanate (pMDI). The best mechanical performance was obtained for the panels glued with pMDI (8% and 10%). The other three adhesives with ratios of 12% and 14% recorded values in the limits imposed by standard EN 312 (2004) for panels of general use.

Another research direction (Dukarska *et al.* 2006) was conducted using mixed wood (W) and rape straw (RS) particles in various ratios for single layer particleboard preparation. Three types of adhesives, *i.e.*, PF (10%), MPF (10%), and pMDI (8%) were used for the experimental panels. Even if the results of mechanical tests were satisfactory for rape straw particles ratio up to 75% and all types of adhesives, water absorption and thickness swelling after 24-h water immersion were not in the upper limits of standard EN 312 (2004).

Dziurka and Mirski (2013) investigated low density panels (350 kg/m^3 to 550 kg/m^3) made from beech (1.7 mm thick) veneered rape straw particleboards using pMDI. Only panels with a higher density (550 kg/m^3) fulfilled all the limits required by EN 312 (2004); the other structures recorded values below accepted limits for bending strength tests. Other boards made from wood chips, rape straw, and expanded polystyrene bonded with MUF resin and having a density of 600 kg/m^3 met the mechanical requirements for boards intended for interior design and furniture (Dziurka *et al.* 2015). Dukarska *et al.* (2017) studied the possibility of using RS particles glued with hybrid pMDI/PF resin in the weight ratio 70:30. Boards with densities between 450 kg/m³ and 650 kg/m³, improved strength properties and water resistance. Three-layer particleboards made in semi-industrial conditions from rape straw (50% of the core) showed enhanced technological properties (Kowaluk *et al.* 2007) in terms of reduced friction forces during the cutting process.

Even if pMDI resins proved to be the most suitable for gluing straws because they are able to absorb these adhesives, creating a good adherence at the bonding interface (Mo *et al.* 2001; Boquillon *et al.* 2004), they have the great disadvantage of being highly toxic if inhaled, thus affecting the respiratory system and causing skin allergies. Long-term exposure to these adhesives causes chronic respiratory diseases and asthma (Tan 2012). Thus, even though isocyanate has the advantage of reducing formaldehyde emissions when it is incorporated as a binder, it has other drawbacks affecting human health, especially when it is used on an industrial scale.

The objective of the present paper is to provide information about the optimum proportional content of rape straw into the single mat configuration of wood-rape particleboard bound with urea-formaldehyde resin (12 wt%) and to make inference on the performance of mechanical properties in correlation with the percentage of rape straw in

the structure and on its influence on the vertical profile density (VPD) and on the morphological structure of the composite. The panels' target density of 640 kg/m³ is similar to that of the particleboards manufactured at the industrial scale. The wood raw materials were provided by a Romanian producer and consist of 70% spruce wood particles and 30% beech wood particles. Since UF resin is still used by particleboard manufacturers on a large scale, the information provided by the present study has a practical application. In an effort to predict the mechanical performance of the composites, this work involved macro- and microscopic investigation of the cross sections of the experimental panels and at the level of the interface between particles, together with VDP analysis.

EXPERIMENTAL

Materials

Mixed beech and spruce wood particles (30% for beech wood and 70% for spruce wood) used for the experimental panels were provided by a Romanian particleboard manufacturer. The moisture content of wood particles were determined by the gravimetric method and ranged from 7.9% to 8.1%. The wood chips sizes were between 5 mm and 20 mm in length, 2 mm and 6 mm in width, and 0.5 mm to 2 mm in thickness. Rape straw stems with moisture content between 11% and 12% were cut into short pieces and crushed by hammer milling. The obtained particles were sieved using a horizontal screen shaker with sieves of 2 mm² x 2 mm², 1 mm² x 1 mm², and 0.5 mm² x 0.5 mm² to remove the dust and to obtain the fractions necessary for the mat. Only particles retained in the 1 mm² x 1 mm² and 0.5 mm² x 0.5 mm² x 0.5 mm² sieves were used to make particleboard. A total of 87.5% of the used rape straw particles remained in the 1 mm² x 1 mm² sieve, and 6% of the particles remained in the 0.5 mm² x 0.5 mm² sieve. The remaining 6.5% of the particles were removed as dust (Fig. 1). The sizes of the rape straw particles varied from 1 mm to 5 mm in length, 0.5 mm to 2 mm in width, and 0.1 mm to 0.5 mm in thickness.



Fig. 1. Rape straw particles used for the experimental particleboards

Based on the particle measurements for rape straw and wood particles, the slenderness ratio (as the ratio of mean values of length and thickness of particles), fatness ratio (as the ratio of mean values of width and thickness of particles), and the width factor (as the ratio of mean values of length and width of particles) were calculated as previously described (Dukarska *et al.* 2017). The results are presented in Table 1.

	Size	es (Mean '	Values)	Sha	pe Analysis	3
Type of Particles	Length (mm)	Width (mm)	Thickness (mm)	Slenderness Ratio	Fatness Ratio	Width Factor
Rapeseed straw	3	1.25	0.75	4	1.7	2.4
Wood	12.5	4	1.25	10	3.2	3.13

Table 1. Sizes (Mean	Values) and S	hape Analysis o	of Rapeseed	Straw Particles
and Wood Particles				

The chemical composition of the rape straw stems showed a lower content of lignin and cellulose compared with solid wood (beech and spruce wood), and an increased content of wax, pentosane, mineral compounds, and ash (Dziurka *et al.* 2005; Szczepkowski *et al.* 2007; Potůček and Milichovský 2011; Huang *et al.* 2016). The higher content of wax in rape straw protects the stalk against water penetration. In particleboard manufacturing, weak adhesion at the particle interface may occur, which limits the internal bond of the particleboard and decreases its water resistance. Crushing the rapeseed straw particles as into fines might reduce these drawbacks.

The urea-formaldehyde (UF) resin (1CL KAS, Viromet SA, Victoria, Romania) with a solid content of $66 \pm 1\%$ was used for the experimental panels. This UF resin is currently used for the particleboard manufacturing on an industrial scale and has a dynamic viscosity of 300 to 700 mPa s at 20 °C and GT reactivity of 50 to 62 s at 100 °C.

Experimental Panels

The target density of the experimental panels was 640 kg/m^3 , which is the average density of the 16-mm particleboard manufactured in industrial conditions from wood particles (70% spruce wood and 30% beech wood). Previous studies have shown that a higher ratio of rape straw particles results in boards with unsatisfactory mechanical performance (Dukarska *et al.* 2006). In the present study, the particleboard manufactured in laboratory conditions had a maximum ratio of 70% rape straw particles in the mat configuration, as seen in Table 2.

UF resin was added to the single-mat configuration at a level of 12 wt%, based on the weight of the particles. A total of 1% ammonium chloride, based on the weight of dry resin, was added as a hardener, and 1% wax (based on dry resin) was added as a hydrophobic substance. Finally, 5% water was added based on the weight of the particles.

	Particle	e Ratio (%)				
Panel Code No.	Rape Straw	Mixed Wood (70% spruce wood and 30% beech wood)	Adhesive (UF) Resin (%)	Press Temperature (°C)	Pressing Time (min)	Press Pressure (bar)
RSP 0	0	100	12	180	6	80
RSP 1	10	90	12	180	6	80
RSP 2	30	70	12	180	6	80
RSP 3	50	50	12	180	6	80
RSP 4	70	30	12	180	6	80

Five types of panel were made, as indicated in Table 2. The panels were prepared under laboratory conditions using a hot press with plates of 450 mm x 450 mm.

The glue mixture was sprayed onto the pre-weighed raw material and blended for 5 min at ambient temperature to obtain a homogenized mixture. The wax was added afterwards, and the particleboards were manually formed in wooden frames whose dimensions are 440 mm x 440 mm x 16 mm. The mat of the experimental panels had initial thicknesses between 40 mm and 50 mm depending on the rape straw participation rate. Boards measuring 400 mm x 400 mm were then hot-pressed at 180 °C for 6 min under 2.5 N/mm² pressure.

After pressing, the particleboards were conditioned at 20 °C and 65% relative humidity for 1 week before evaluating the physical and mechanical properties. The particleboards were first trimmed to avoid edge defects to a final size of 390 mm x 390 x 16 mm. Three replicate panels were made for each board type, and specimens for mechanical and physical testing were cut from each particleboard type. The numbers and sizes of the specimens used for tests were according to the following standards: EN 317 (1993) for TS and WA, EN 310 (1993) for BS and MOE, and EN 319 (1993) for IB perpendicular to the plane of the board. The specimens used for X-ray density profile analysis were cut at the sizes of 50 mm x 50 mm (Fig. 2).



Fig. 2. Samples cut for testing the mechanical and physical properties of the experimental panels

The performance of the particleboards was analyzed by comparing the results with limits imposed by EN 312 (2004) standard requirements in terms of bending strength (BS), internal bond (IB), modulus of elasticity (MOE), water absorption (WA), and thickness swelling (TS).

Physical Testing

The physical properties of WA and TS were determined after 24 h of water immersion (EN 317 1993). Five specimens from each particleboard type were used for WA and TS tests. WA and TS tests were performed by submerging the specimens in water at room temperature (20 $^{\circ}$ C) for 24 h, and calculated based on the weight and thickness measurements before and after immersion in water. The mean values were reported.

The density profiles were provided on 50 mm x 50 mm test specimens using X-ray density profile analyzer DPX300 (IMAL, San Damaso, Italy). Vertical density profiles were measured on six specimens with sizes of 50 mm x 50 mm x 16 mm.

Morphological Analysis

Scanning of the samples was performed to analyse morphological structure of particleboard composites. The scanned specimens were taken from cross section of experimental panels. Samples had the dimensions (L x W x T) of 50 mm x 16 mm x 5 mm. A HP Scanjet 7650 scanner, China was employed for macroscopic study. The samples were scanned with the highest resolution of 4800 dpi in order to achieve maximum magnification of textural and morphological characteristics of the panels on their thicknesses. The scanned images were afterwards cropped and magnified, so to achieve a clear textural representation of the composites. In addition, the panels' microstructure was investigated using a 90x magnification Optika SZM-2 ocular stereomicroscope, Italy. The microscope is equipped with a high resolution digital video camera and a specific vision software package. Images with 20x magnification were captured, showing the internal defects of the investigated structures.

Mechanical Testing

The BS, MOE (EN 310 1993), and IB perpendicular to the plane of the board (EN 319 1993) were determined using a Zwick/Roell Z010 universal testing machine (Ulm, Germany). Six specimens cut from each type of panel. The specimen lengths for MOE and BS tests were calculated depending on the panel thickness, according to EN 310 (1993). The mean values of the measurements were recorded.

RESULTS AND DISCUSSION

Physical testing

Density profiles of the thickness of the tested specimens showed differences between median surface and top/bottom surfaces. Selections of the density profile analyzed along the thickness of the specimens obtained from each type of investigated particleboard are presented in Fig. 3. The density profiles were generated by the of X-ray density profile analyzer software along the thickness of 16 mm. Figure 3 shows the influence of the rape straw share on the density profile of the 16-mm particleboard. Similar profiles were obtained by Belini *et al.* (2014) for composites made of bagasse and eucalyptus. They detected decreased and inhomogeneous density in the central region along the thickness for panels with 75% and 100% bagasse, indicative of incomplete resin cure, or improper particle size and anatomical structure of the particles in the inner panel, or changes in the press cycle variables. Hunt *et al.* (2017) explained that an M-shape graph is the typical profile for both medium and high-target particleboard, with

higher peak density regions to the outer faces and approximately flat in the core (as for profile densities of RSP 0 and RSP 1 in Fig. 3). On the other hand, U-shape profile (as for RSP 2, RSP 3 and especially RSP 4) is explained by decreasing pressure in the core as the moisture left and by a densification process in the face region within which the particles were softened by steam. Hunt *et al.* (2017) showed that there is a correlation between the vertical density profile and IB test and most failures occur in the low density core region during this test.

Generally, up to 4 mm from the surface areas, the density reached top values between 660 kg/m³ to 700 kg/m³ for RSP 1, 700 kg/m³ to 750 kg/m³ for RSP 2 and RSP 3, and 750 kg/m³ to 800 kg/m³ for RSP 4. In contrast, in the center area of the specimens, fluctuating density profiles were reported with values between 500 kg/m³ for the higher ratios of rape straw particles in the mat (70%) and 580 kg/m³ for the lowest ratio (10%).

A uniform structure was observed for RSP 0 specimens, where the differences between the top values and the minimum ones were no more than 40 kg/m³, compared with 120 kg/m³ to 300 kg/m³ for the other specimens. Given the density profiles, it was expected that specimens subjected to internal bond test would break in the core area but that particleboards with a lower ratio of rape straw particles would demonstrate higher strength.





Fig. 3. Influence of rape straw-to-wood ratio on the density of particleboard along the thickness

The evolution of thickness swelling was recorded after 2 h, 4 h, 6 h, and 24 h of sample immersion into the distilled water at 20 °C and is presented in Fig. 4. The thickness swelling dynamic was higher in the first four hours of immersion and remained almost constant between 6 h to 24 h of sample water immersion. The WA and TS values at 24 h of water immersion are shown in Table 3. With increased of rape straw content in the particleboards, the TS and WA increased. The same trend was noticed for mixed wood-rape straw particleboard, where melamine urea phenol-formaldehyde and phenol formaldehyde resins were used as bonding agents (Dukarska et al. 2006). In that case, TS varied between 25% and 51%, and WA recorded values between 73% and 109% for 24 h immersion in water. In comparison, the values obtained in the present research for mixed wood-rape straw particleboard were better for TS (18.4% to 29.3%) and similar for WA (76.9% to 112.3%), as shown in Table 3. The increased rape straw content in the particleboard decreased its water resistance. Thus, RSP 1 had better water resistance than RSP 2, RSP 3, and RSP 4, but was lower than RSP 0, which was made only from mixed wood. A polynomial model fit in R was used to model the effects of rape straw content on the WA and TS, (Fig. 5), with $R^2 > 0.97$.



Fig. 4. Evolution of thickness for 24 h of samples immersion into the water



Fig. 5. Relationship between the rape straw share rate and WA/ TS

Morphological analysis

The morphological examination of the composites on their thicknesses showed different microstructure due to species ratio of single layer particleboard. The macrostructure analysis of the composites was carried out on the scanned images shown in Fig. 6. The marked zones in the images represent gaps in the structure.

From the images in Fig. 6, it can be seen that the increase rate of rape straw particles in the panels' structure affected the interfacial bonding between particles in boards. The red marked zones show the gaps and fracture zones in the structure. They extend as surface and magnitude with the increase of rape straw (RS) ratio.

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Fig. 6. Scanned images showing the morphology of the panels with the increasing RS ratio; a. 100% W; b. 10% RS; c. 30% RS; d. 50% RS; e. 70% RS. Red circles represent gaps in the structure

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Fig. 7. Particleboards' microstructure showing the poor adherence between wood-rape straw particles for the composites (as indicated by yellow circles) with higher participation rate of RS particles; a. 100% W; b. 10% RS; c. 30% RS; d. 50% RS; e. 70% RS

Thus, the increase of the RS ratio when using UF adhesives resulted in decreased adherence between the surfaces of wood-rape straw particles from the core zone of the particleboard composites. The microscopic structure of the investigated panels, as images with 20x magnification, are presented in Fig. 7. The marked zones in the images represent gaps and insufficient bonding areas between wood particles and rapeseed straws.

Mechanical testing

The experimental results of mechanical testing for BS, MOE, and IB are presented in Table 3. The results are compared with the requirements of the EN 312 (2004) standard for panels designed for general purpose (P1), furniture manufacturing (P2), and wet environments (P3).

Panel code no.	WA	TS	BS	MOE	IB
	24 h (%)	24 h (%)	(N/mm²)	(N/mm²)	(N/mm²)
RSP 0	57.8	17.8	13.8	3728	0.39
	(1.3)	(0.14)	(0.4)	(62.1)	(0.05)
RSP 1	76.9	18.4	13.5	3720	0.35
	(1.99)	(1.04)	(1.76)	(537)	(0.08)
RSP 2	87.8	19.1	12.7	3640	0.30
	(5.55)	(0.36)	(0.88)	(253)	(0.03)
RSP 3	107.2	23.4	9.1	2760	0.25
	(8.70)	(0.29)	(0.45)	(122)	(0.04)
RSP 4	112.3	29.3	6.3	1380	0.16
	(6.29)	(0.45)	(0.18)	(32.5)	(0.09)
EN 312 (2004)		14 ^c	11.5 ^a 13.0 ^b	1600 ^b	0.24 ^a 0.35 ^b
^a Minimum ^b Minimum ^c Maximum	value for panels value for panels value for panels	designed for gen designed for furn designed for wet	eral purpose (P1) iture manufacturi environment (P3	ng (P2) 3)	
The values	in parenthesis re	present the stand	lard deviation		

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The ANOVA analysis of variance shows that all physical (WA, TS) and mechanical (BS, MOE, IB) were statistically significant (p-value < 0.05) with the variation of rape straw content in the structure of particleboards. Among them, the effect of IB was the least significant (p-value = 0.000125) compared to the other factors with p-value < 0.0001.

As the results in Table 3 show, the presence of rape straw particles in the mat had a negative influence on the mechanical properties of the particleboard. The overall mechanical testing results of RSP 1 met the requirements of the EN 312 (2004) standard, both for P1 and P2 panels; however, the IB mean value for this structure was equal to the upper limit imposed by the above-mentioned standard. Furthermore, the RSP 2 mechanical performance complied with the EN 312 (2004) standard requirements for P1 (panels designed for general purpose). RSP 3 and RSP 4 met neither the EN 312 (2004) standard requirements nor for P1 panels, as seen in Table 3.

The second order polynomial regression model with an R-square value over 0.98 appeared to fit the experimental data for the BS, MOE, and IB depending on the rape straw content (Fig. 8). A more abrupt decline was noticed for the polynomial curve of BS in Fig. 8.



Fig. 8. Variation of mechanical properties (BS, MOE, IB) depending on the content of rapeseed straw

The correlation between the microstructure, vertical profile density, and the behavior of the samples to mechanical testing was established by the way the samples have been broken during IB test (Fig. 9)



Fig. 9. Rupture zones of the samples during IB test

As can be seen in Fig. 9, RSP 1 specimens failed close to the surface. This was correlated to the vertical density profile, where the peak density was reached at 2 mm from the surface area, and near the surface the density was lower. As can be seen in the images from Fig. 6 and Fig. 7 and on the profile densities of RSP 2 and RSP 3 in Fig. 3, low density and porous structure occurred closer to the core, as confirmed by IB test (Fig. 9). For the U -shaped density profile with low core density of RSP 4, IB tests confirmed that specimens broke in the core region.

An explanation of the poor mechanical performance of the composites with high percentage of rape straw may be also the chemical composition of rape straw. As found in the literature (Winandy and Rowell 1984; Sari *et al.* 2012), the chemical composition of wood is one of the factors influencing mechanical properties of particleboard. Thus, cellulose is responsible for strength in the wood fiber because of its high degree of polymerization and linear orientation, so high cellulose content resulted in superior mechanical properties. In contrast, high hemicelluloses content is detrimental to the mechanical properties. Based on these assumptions, lower content of cellulose in rape straw, namely 37.6% compared to 54.1% for spruce wood (Dziurka *et al.* 2005) and 49.4% for beech wood (Szczepkowski *et al.* 2007) could alter the strength of composites for a higher content of rape straw particles (30%, 50% or 70%). In addition, the higher content of hemicellulose in rape straw compared to 23.4% (Dziurka *et al.* 2005) is detrimental to the mechanical performance of

the panel. On the other hand, olefin substances contained by rape straws play an important role in the determination of bond quality.

The sizes and shapes of particles also contribute to the mechanical properties of the composites. Considering the theory presented by Sackey *et al.* (2008), the better flexural properties of the experimental panels having an increased wood chips proportional content (from RSP 4 to RSP 0) may be explained by the slenderness ratio (length-to thickness ratio) which is 2.5 times higher in case of wood particles (Table 1) compared to rape straw particles. Yunus *et al.* (2018) also state that bigger particle size contributes to better mechanical properties. Other theories show that the decrease of the particle size (Boquillon *et al.* 2004) or for thicker and shorter flakes (*i.e.*, lower slenderness ratio and higher width ratio) (Sackey *et al.* 2008) the contact area between the resin and the straw particles increase and as a consequence, IB increases. For the case of experimental panels with increased participation rate of rape straw particles (RSP 0 to RSP 4) IB decreased, even if the first condition of lower slenderness ratio of these particles was fulfilled. For this case other factors (chemical, adhesive type and its share rate) affect the adherence between particles and resin.

CONCLUSIONS

- 1. The mechanical performance of the particleboard in terms of bending strength, modulus of elasticity, and internal bond decreased with the increasing rape straw content. A more pronounced fall was noticed for bending strength.
- 2. The single mat particleboard with a density of 640 kg/m³ and structure share rates of 90% mixed wood and 10% rape straw particles, which were bonded with ureaformaldehyde resin, had an appropriate mechanical strength according to the EN 312 (2004) standard to be used for panels designed for furniture manufacturing (P2).
- 3. Study of the morphological structure and of the vertical density profile show that the increase rate of rape straw particles in the panels' structure affects the interfacial bonding between particles in the core region, thus affecting the mechanical performance of the boards.
- 4. Both thickness swelling and water absorption after 24 h of particleboard water immersion increased with increasing rape straw content with polynomial regressions, whilst the bending strength, modulus of elasticity, and internal bond decreased as polynomial regressions.

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