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Fired clay bricks using agricultural biomass wastes: Study and characterization

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HIGHLIGHTS

- Wheat straw, sunflower seed cake and olive stone flour have been incorporated into clay matrix.
- The influence of the grinding and incorporation rate of the additives have been studied.
- Physical, mechanical and thermal properties of the fired brick have been affected.

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ABSTRACT

The main objective of this study is to investigate the effects of the incorporation of renewable pore forming agents on the properties of fired bricks. Different additives have been studied (wheat straw, sunflower seed cake and olive stone flour) at different grinding and incorporation rate.

Physical properties such as linear shrinkage, loss on ignition, bulk porosity, water absorption and bulk density have been measured. Mechanical and thermal performances have also been characterized. The incorporation of 4 wt.% of sunflower seed cake, with the lowest grinding, leads to the best compromise between mechanical and thermal results compared to the reference brick.

1. Introduction

Based on a simple manufacturing process and the use of cheap and abundant raw materials (clay, sand and water), clay bricks are one of the most used building materials. Indeed, they present interesting mechanical and thermal properties [1]. However, with the recent development of more reliable materials, bricks come across technical limits because of their weight and limited thermal resistance.

In a context of sustainability and with the always more restric tive environmental regulations, environmentally friendly materials recycling wastes and saving energy have to be developed. It has been proven in the literature [2 4] that the incorporation of by products or wastes is an innovative and efficient way to generate pores in fired bricks. The described pore forming agents can be divided into two categories: those issued from renewable resources (for example rice straw [5], processed waste tea [6], sawdust [7]) and those from mineral resources (such as slags [8] or marble residues [9]). It was therein shown that a small amount of additives (generally below 10 wt.%), that bums during the firing process, led to an increase of the brick porosity and so a decrease of the thermal conductivity. However, it was also observed that this creation of pores generally caused a significant decrease of the mechanical performances [10]. Compromises between mechanical

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and thermal properties have thus to be made in order to produce a competitive building material. Numerous pore forming agents have been tested in the literature, most of the time in order to lighten the final brick, the thermal aspect being left aside.

In this work, we focused on the incorporation of new additives such as wheat straw, sunflower seed cake and olive stone flour which were chosen for their low cost, availability and close location. The last two materials were never incorporated into clay bricks formulation; the sunflower seed cake being mostly used for animal feeding or to develop insulation fiberboards [11] or injection moldable composite materials [12]. The olive stone flour was, on the other hand, used for producing thermoplastics [13]. Wheat straw has been once used as pore forming agent in a former project of our laboratory [14] but the results obtained were not enough optimized.

After characterizing the different raw materials (vegetable matter and clay), the influence of the nature of the pore forming agent, their grinding and incorporation rate were studied. Finally, the physical (linear shrinkage, loss on ignition, porosity, water absorption and bulk density), mechanical (bending strength) and thermal properties of the new developed bricks were evaluated.

2. Materials and methods

2.1. Characterization of the brick raw materials

2.1.1. Clay characterization

The clay mixture used in this study was provided by TERREAL (Castelnaudary, France).

The elementary compositions were measured through energy dispersive X-ray spectroscopy (EDX, 15 kV and 10 nA).

2.1.2. Agricultural wastes characterization

The three types of biomasses used in this study (wheat straw, sunflower seed cake and olive stone flour) were selected as they are locally produced, readily available and cheap. Furthermore, their distinct composition and grinding should induce different behaviors during the firing process.

The wheat straw and sunflower seed cake were provided by the agricultural cooperative ARTERRIS (Castelnaudary, France) and the olive stone flour by BARDON ETS (Le Muy, France).

The biomasses were first chemically characterized. The dry matter content was determined according to the French standard NF V 03-903. The mineral content was determined according to the French standard NF V 03-322. The fiber content (cellulose, hemicelluloses and lignin) was determined using the ADF-NDF method [15–17]. The protein content was determined using the Kjeldahl method according to the French standard NF V 18-100 with a multiplying factor of 6.25 for the nitrogen percentage. The lipid content was determined after a Soxhlet extraction using cyclohexane, during 6 h, according to the French standard NF V 03-908. All the characterizations were performed in triplicate.

The thermogravimetric analysis (TGA) of the vegetable matters was performed using a simultaneous thermal analyzer (NETZSCH STA 449 F3 Jupiter). The measure consists into reporting the weight loss of the sample of about 200–500 mg, placed into a cylindrical crucible, with an increase of temperature up to 1100 °C at a rate of 10 °C/min. The results were then derived using the PeakFit software to obtain the differential curve of the thermogravimetric analysis (DTG), in order to identify the decomposition occurring in the samples.

The sorption and desorption behaviors of all the agricultural wastes were observed using a dynamic vapor sorption (DVS) Advantage System from Surface Measurement Systems (Alperton, UK). The apparatus uses an ultra sensitive balance capable of measuring changes in sample mass as low as 0.1 μg. Samples were equilibrated at a constant temperature and different relative humidities (from 0% to 90%). The changes in relative humidity were induced using mixtures of dry and moisture-saturated nitrogen flowing over the sample. At the beginning of each measurement, samples were dried under dry nitrogen. From the complete moisture sorption and desorption profile an isotherm was plotted.

The water absorption, also called swelling *ratio*, of the vegetable matters was also determined by soaking the biomass into an excess of water overnight and measuring the relative increase of the solid volume when saturated in water.

2.2. Samples preparation

The clay was first ground in order to obtain a powder with particles of about 3 mm. Wheat straw and sunflower seed cake were crushed and sieved at different grindings: <0.5 mm and [0.5,1.0] mm. The olive stone flour was industrially ground at about 50 μm. Different amounts (4 and 8 wt.%) of additives were then mixed with clay in a rolling mill to enhance homogeneity.

The required quantity of water (up to 22.2 wt.% depending on the formula) was added to obtain the desired humidity and plasticity that are necessary to avoid defects onto the structure during the process.

The samples were then molded by extrusion process in the form of tablets ($175 \times 79 \times 17$ mm^3), dried up to 105 °C and finally fired up to 920 °C for 1 h, according to the industrial recommendations.

Samples were prepared and designated as Ref. for the bricks without waste and x-ByP-g for the mixtures with x the content of additives incorporated (in wt.%), ByP the by-product used (ByP = WS (wheat straw), ByP = SSC (sunflower seed cake) and ByP = OSF (olive stone flour)) and g the grinding of the biomass (in mm).

The sample composition is presented in Table 1.

2.3. Characterization of the bricks

The physical (linear shrinkage, loss on ignition, density, water absorption and porosity), mechanical (bending strengths) and thermal properties of the obtained clay bricks were determined.

Linear shrinkage was determined by measuring the length of the sample before and after drying using a caliper according to the standard ASTM C210-95. Loss on ignition was determined by measuring the mass loss of the sample between the drying and firing steps. Water absorption of these lightweight bricks was determined using the standard procedure ASTM C 373-88. The samples were dried at 110 °C for 24 h and weighed to constant mass. They were then cooled for 24 h and totally immersed in water. After soaking for 24 h, they were dried and reweighed to constant mass.

The bulk porosity and saturated density were determined according to the test procedure recommended by Hornain et al. [18] by means of water saturation under vacuum.

Thermal conductivity was obtained through a heat flux meter method. This method followed the standards ASTM C518, ISO 8301 and NF EN 12667. The measurement area was 60x40 mm^2 with a thickness of the sample greater than 10 mm. This apparatus produced a temperature gradient along the thickness of the sample and measured heat flux that gave through the software the thermal effusivity. Thermal conductivity was deduced using the formula: $\lambda \ \frac{E^2}{\rho C_p}$ (W/m K) with ρ the bulk density (kg/m^3) and C_p the specific heat capacity (J/kg C).

All the characterizations were performed on 6 samples; the coefficient of variation of all the obtained values, defined as the *ratio* of the standard deviation to the mean, is verified to be prior to 5%, showing the accuracy of the presented data.

3. Measurements and results

3.1. Clay characterization

In Table 2 is reported the chemical composition of the fired clay. From these data, it is apparent that this clay is mainly composed of quartz (SiO_2), aluminum oxide (Al_2O_3) and hematite (Fe_2O_3). Calcium, potassium and magnesium oxides are present but to a less extent. Traces of titanium, sodium and phosphorus oxides were also observed.

Table 1
Sample compositions.

Sample	Ref.	4-WS-<0.5	8-WS-<0.5	4-WS-0.5/1	4-SSC-<0.5	4-SSC-0.5/1	4-OSF	8-OSF
WS (wt.%)	–	4	8	4	–	–	–	–
SSC (wt.%)	–	–	–	–	4	4	–	–
OSF (wt.%)	–	–	–	–	–	–	4	8
Water (wt.%)	15.9	19.1	22.2	19.5	20.3	19.8	17.3	19.0

Table 2
Chemical composition of the fired clay (in wt.%).

	SiO_2	Al_2O_3	Fe_2O_3	CaO	K_2O	MgO	TiO_2	Na_2O	P_2O_5
Fired clay	44.7	22.7	10.8	6.7	6.6	6.1	1.9	0.4	0.1

Table 3
Composition of the biomasses.

	Wheat straw	Sunflower seed cake	Olive stone flour
Dry matter (%)	90.86 ± 0.07	90.94 ± 0.01	94.56 ± 0.04
Proteins[a] (%)	2.18 ± 0.28	34.14 ± 0.18	1.17 ± 0.01
Cellulose[a] (%)	42.14 ± 1.07	27.8 ± 1.5	36.80 ± 0.08
Hemicellulose[a] (%)	11.88 ± 0.75	11.0 ± 3.8	25.59 ± 0.08
Lignin[a] (%)	28.92 ± 0.88	7.4 ± 0.4	31.16 ± 0.23
Lipids[a] (%)	4.57 ± 0.78	2.63 ± 0.23	3.52 ± 0.38
Ash[a] (%)	8.89 ± 0.22	7.19 ± 0.87	0.99 ± 0.03

[a] Expressed on the recovered dry matter.

3.2. Biomass characterization

The chemical composition of the agricultural by products was determined using the protocol described in Section 2.1.2. The results obtained for these additives are shown in Table 3.

It can be seen from these data that these three biomasses pre sent significantly different compositions: the wheat straw and the olive stone flour contain more than 80% of fibers [19 21]. Compared to the olive stone flour which is rich in cellulose (36.80%), hemicellulose (25.59%) and lignin (31.16%), the wheat straw contains less hemicellulose and more cellulose (respectively 11.88% and 42.14%). As for the studied sunflower seed cake, it is mainly composed of proteins (34.14%) and cellulose (27.8%) [22,23]. The three matters also contain various amounts of mineral matter (from 0.99% for the olive stone flour to 8.89% for the wheat straw) that will be residual once incorporated into the fired clay brick formulation.

Thermogravimetric analyses were then carried out on these vegetable materials (Fig. 1).

Various behaviors were observed for the thermal decomposi tion of the biomasses: the wheat straw presents the fastest degra dation, which starts at 240 °C to reach a maximum weight loss of 87% at 570 °C. The degradation of the olive stone flour and the sun flower seed cake is slower, as their weight loss begins to level off around 1000 °C.

The DTG analysis showed a first peak, around 100 115 °C (according the biomass), which represents the evaporation of the free water contained in the vegetable samples. The major peak is observed around 250 °C and stands for the thermal decomposition of the cellulose, with its depolymerization into anhydrosugar derivatives and their volatilization [24]. The biggest peak is obtained for wheat straw, whereas the smallest one corresponds to the sunflower seed cake. These results are in total accordance with the chemical composition of these samples (Table 3), with the wheat straw which is rich in cellulose (42.14%) compared to the seed cake (27.8%). Besides, a small shoulder can be observed before the cellulosic peak. It is characteristic of the decomposition of hemicellulose, wax or even pectins which are thermally less stable than cellulose [25,26]. The decomposition of lignin occurs at higher temperature, between 250 and 500 °C [24].

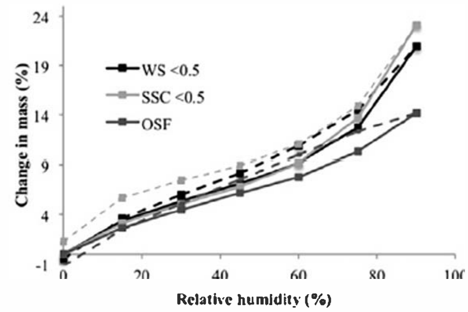

Fig. 2. Sorption and desorption behavior of the three vegetable matter at the lowest grinding (WS: wheat straw, SSC: sunflower seed cake and OSF: olive stone flour); the continuous lines corresponding to the sorption and the discontinuous lines to the desorption.

The ability of these additives to absorb and release water was then studied (Fig. 2). Indeed, this parameter is important as it has a direct impact on the ideal formulation for the preparation of the desired fired brick. As a matter of fact, water is added to the mixture clay vegetable matter in order to obtain the proper plasticity before the extrusion process.

The water sorption and desorption curves that are plotted in Fig. 2, show that the sunflower seed cake presents a more impor tant water content than wheat straw or even olive stone flour. These data are in agreement with the swelling *ratios* that were cal culated (Table 4): the biggest swelling *ratio* (170%) was obtained for the sunflower seed cake whereas the smallest one was obtained for the olive stone flour (120%).

3.3. Bricks characterization

The newly developed bricks were then dried and fired. Several properties were measured and compared to the reference without additive.

The linear shrinkage (LS), which represents the change in dimensions of the material before and after drying, is a critical property. Indeed, a large contraction of the sample could create tension and breakage [27]. These results are reported in Table 5.

For all samples, the addition of wastes decreases the linear shrinkage, i.e. the loss in dimensions for samples with additives is smaller than for the reference with only clay (5.8% for the refer ence against 3.3 5.5% for the porous products). The incorporation of pore forming agent, and so the decrease of the amount of clay,

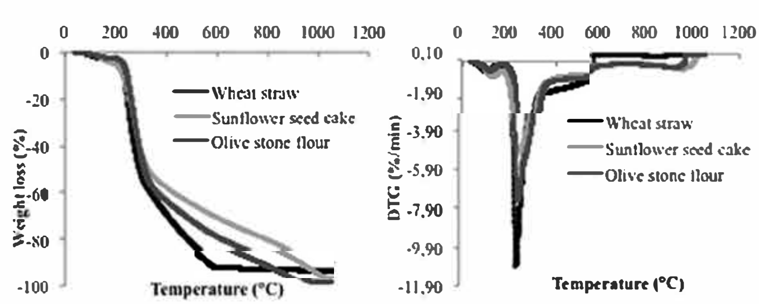

Fig. 1. Thermogravimetric analysis (TGA) of the by-products used in this study (wheat straw, sunflower seed cake and olive stone flour).

Table 4
Swelling *ratio* of the agricultural wastes.

Sample	WS < 0.5	SSC < 0.5	OSF
Swelling *ratio* (%)	145 ± 5	170 ± 1	120 ± 8

leads to a decrease of linear shrinkage [28]. The material is then less contracted with the incorporation of biomass in comparison to a reference sample containing only clay and sand.

These results are consistent with the work of Faria et al. [29] or Görhan and Şimşek [28]. The vegetable material acts, in the matrix, as a load or an inert; it decreases the plasticity mixtures and thus the contact between the clay particles as well as the sand, and therefore, reduces the linear drying shrinkage in our case, but also the total one of the whole process [30].

This linear shrinkage characterizes the evaporation of water out of the matrix upon drying. It is greatly related to the amount of water required for the preparation of the formulation. Indeed, sam ples containing sunflower seed cake (4 SSC <0.5 and 4 SSC 0.5/1) present the most important shrinkage but also the higher water content (Table 1), in comparison with the other samples with the same incorporation rate, as also shown by the work of Saiah et al. [14]. As the sunflower seed cake presents the biggest swelling *ratio* (Table 4), a larger amount of water must be added during shaping to achieve comparable plasticity of clay formulations con taining the two other additives. Indeed, the water will preferen tially be retained by the biomass and less by the clay particles.

The loss on ignition (LoI) representing the weight loss of the sample after firing is presented in Table 5. The incorporation of wastes into the system increases this loss as vegetable matter decomposes at high temperature between 570 and 1000 °C (as pre sented on the TGA results on Fig. 1) with dehydroxylation and decarbonatation and so creation of pores. Only the mineral part of the biomass, representing 1 to 10 wt.%, remains in the matrix after firing.

The impact of the incorporation rate can be highlighted: for example, for the incorporation of wheat straw at a grinding inferior to 0.5 mm, a loss on ignition of 10.5% was obtained for an incorpo ration of 4 wt.%, compared to a loss on ignition of 13.5% for an incorporation of 8 wt.%. We can thus conclude that the more waste in the formulation, the more important weight loss.

As for the grinding and the nature of the by product, they do not have much impact on the result as a value of about 10% is obtained for the three wastes at 4 wt.%. Noteworthy, the results obtained on the different samples are still under the recommended industrial value of 15% [31].

The incorporation of pore forming agents into the clay matrix leads to changes on other characteristic physical properties of the bricks: porosity, water absorption (WA) and bulk density (BD) (Table 6).

Compared to the value of the reference sample, the porosity increases with the incorporation of vegetable matter: a value of 25.6% is obtained for the sample Ref. against a value of 31.6% for 4 SSC <0.5 and 43.5% for 8 WS <0.5. Bulk density (BD) changes from 1.95 g/cm^3 for the reference to values between 1.46 and 1.77 g/cm^3 for respectively the samples 8 WS <0.5 and 4 OSF. Regarding water absorption (WA), the values increase with the formation of pores: the sample Ref. having a value of 12.5% and porous samples values between 17.8% and 30.0%.

Adding vegetable wastes into the structure leads to an increase of the porosity and the water absorption, correlated to a decrease of the bulk density of the samples, leading to a lightening of the material. Indeed, during the manufacturing process especially dry ing and firing steps, biomass particles breakdown leaving pores in the clay matrix, which results in an increase of the bulk porosity. Because of this voids creation, the material is more likely to absorb water, depending on the way particles are connected to one another, corresponding to the increase of water absorption. But this increase of porosity is also related to a decrease of the bulk density of the material, as air is lighter than clay.

According to the values presented in Table 6, it can be noticed that the incorporation rate, within the limits of our experiments, induces more important modifications than the nature or even the grinding of the added waste. Indeed, adding 4 wt.% of any bio mass at any grinding results in an increase of about 29 49% for the porosity, 42 76% for the water absorption and 9% to 16% for the bulk density. In comparison, the incorporation of 8 wt.% of biomass leads to much larger deviations with an increase of about 60 80% for the porosity, 93 140% for the water absorption and 17% to 25% for the bulk density.

Focusing only on the nature of the additive, at similar incorpo ration rate and grinding, it appears that samples with wheat straw have a higher porosity and water absorption. This seems to be explained by the particle size but also the faster decomposition of straw compared to other biomasses (Fig. 1). It is recommended that the additive decomposes before quartz point (at 573 °C) in order to avoid weakening the material during the transformation of clay into ceramic.

As the major aim of this study is to produce an interesting light weight brick with high thermal and mechanical performances, the mechanical and thermal characteristics of these new developed bricks were finally analyzed (Table 7).

Table 5
Linear shrinkage (LS) and loss of ignition (LoI) of the bricks with agricultural biomass wastes (Ref. for the bricks without waste and *x*-ByP-*g* for the mixtures with x the content of additives incorporated (in wt.%), ByP the by-product used (ByP = WS (wheat straw), ByP = SSC (sunflower seed cake) and ByP = OSF (olive stone flour)) and *g* the grinding of the biomass (in mm)).

Sample	Ref.	4-WS-<0.5	8-WS-<0.5	4-WS-0.5/1	4-SSC-<0.5	4-SSC-0.5/1	4-OSF	8-OSF
LS (%)	5.8	4.4	3.3	4.1	5.5	5.4	5.2	5.3
LoI (%)	7.7	10.5	13.5	10.6	10.5	10.6	10.8	14.5

Table 6
Physical properties of the bricks with agricultural biomass wastes (Ref. for the bricks without waste and *x*-ByP-*g* for the mixtures with *x* the content of additives incorporated (in wt.%), ByP the by-product used (ByP = WS (wheat straw), ByP = SSC (sunflower seed cake) and ByP = OSF (olive stone flour)) and *g* the grinding of the biomass (in mm)).

Sample	Ref.	4-WS-<0.5	8-WS-<0.5	4-WS-0.5/1	4-SSC-<0.5	4-SSC-0.5/1	4-OSF	8-OSF
Porosity (%)	25.6	35.2	43.5	35.9	31.6	33.2	32.7	38.9
BD (g/cm^3)	1.95	1.69	1.46	1.64	1.7	1.7	1.77	1.62
WA (%)	12.5	20.6	30	22	19	19.5	17.8	24.1

Table 7
Mechanical and thermal properties of bricks with agricultural biomass wastes (Ref. for the bricks without waste and *x*-ByP-*g* for the mixtures with *x* the content of additives incorporated (in wt.%), ByP the by-product used (ByP = WS (wheat straw), ByP = SSC (sunflower seed cake) and ByP = OSF (olive stone flour)) and *g* the grinding of the biomass (in mm)).

Sample	Ref.	4-WS-<0.5	8-WS-<0.5	4-WS-0.5/1	4-SSC-<0.5	4-SSC-0.5/1	4-OSF	8-OSF
Bending (MPa)	12.1	9.9	5.3	9.2	10.1	10.8	10.9	7.6
ThC. (W/m K)	0.51	0.32	0.28	0.3	0.2	0.3	0.38	0.33

As a regard to the mechanical results, a decrease in the bending stress can be observed for all samples. If most of the results are rea sonable, some values (for 8 WS<0.5,4 WS 0.5/1 and 8 OSF) are still lower than the value of 10 MPa recommended by the industrial standards. In these cases, corresponding samples may be consid ered too fragile to be used on an industrial scale.

Regarding thermal performance, the thermal conductivity was reduced for all samples, with the addition of vegetable materials with values ranging between 0.2 and 0.38 W/m K, compared with 0.51 W/m K for the reference (without additive), which represents a promising diminution of 25 60%.

The properties influencing the thermal conductivity are mostly the bulk density and porosity [32]. From the results presented in Table 6, it could be assumed that the sample with the highest porosity and the lowest density (that is to say, the sample 8 WS <0.5) should be one that would have the highest insulation performance, and so the lowest thermal conductivity. However, the most insulating sample is 4 SSC <0.5. This result, contradic tory, seems to indicate that the porosity we measured, also called porosity accessible to water, does not lead to a complete measure ment of the total porosity of the sample, that includes for example closed porosity.

Moreover, it seems that beyond the bulk density, the miner alogical composition of the samples and the nature of the pores, their size or distribution, have considerable influence [33 35].

Those data show that a compromise has to be found to develop the ideal product exhibiting high thermal conductivity and bend ing strength. According to Table 7, the best choice is to incorporate into the clay matrix 4 wt.% of sunflower seed cake at the lowest grinding (<0.5 mm) leading to a brick with a thermal conductivity of 0.2 W/m K and a bending strength of 10.1 MPa.

Noteworthy, the incorporation of vegetable matter also led to the formation of black cores. The presence of these darker areas can be explained by the important rise of temperature and libera tion of carbon dioxide, due to the decomposition of vegetable mat ter leading a reductive media [14,36].

4. Conclusion

Nowadays, most of the agricultural wastes are used for live stock feeding or to provide energy (combustion, biofuels, methani sation...). Despite these applications, a large part of biomasses are unmanaged. This work consists into incorporating vegetable mat ters into clay bricks, one of the most common building materials, to produce lightweight and environmentally friendly products.

Wheat straw, sunflower seed cake and olive stone flour were experimented because of their cost, availability and proximity. The influence of nature, grinding and incorporation rate was stud ied. Technological tests realized on the developed fired clay bricks showed the impact of the additives upon the properties of the material: an increase of the porosity, water absorption and thermal insulation was observed, correlated with a decrease of the bulk density and the bending strength. Compromises have thus to be found in order to produce more efficient products with high ther mal and mechanical performances.

In our case, the best compromise is to incorporate 4 wt.% of sun flower seed cake, with the lowest grinding, leading to an increase of 23% of porosity and decreases of 17% of the bending strength and 61% of the thermal conductivity.

The reuse of these wastes clearly presents several advantages whether it is on an technological aspect or on an economical point of view: it leads for example to a reduction of the costs due to the use of wastes by substitution of the clay matter and a reduction of transportation costs due to the production of lighter products. A life cycle assessment is currently realized to determine the envi ronmental impacts of these new matters and will be published in due course.

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