

*Full Length Research Paper*

# A new pozzolanic material for cement industry: Bamboo leaf ash

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**Bamboo leaf fired in an open atmosphere and then heated at 600°C for 2 h in a furnace was found to be an amorphous material containing amorphous silica. The ash was characterized by chemical analysis, powder X-ray diffraction and SEM techniques. Reactions of the ash with calcium hydroxide showed it to be pozzolanic in nature. The pozzolanic reactivity increased with time and temperature. When 20 wt% bamboo leaf ash was mixed with Portland cement, the compressive strength at 28 days of hydration was comparable to that without ash. This was due to pozzolanic reactions. The mechanism of pozzolanic reactivity has been discussed.**

**Keywords:** Bamboo leaf ash, Pozzolana, Portland cement, Hydration, Calcium hydroxide

## INTRODUCTION

In cement industries, continuous attempts are being made (i) to reduce the cost of production of Portland cement, (ii) to reduce the consumption of the raw materials, (iii) to protect the environment and (iv) to enhance the quality of cement. One way is to use certain low cost materials for partial replacement of Portland cement clinker. Low cost materials used are industrial and agricultural by-products (wastes). Mixture of Portland cement and the above by-products are known as 'blended cements' or 'composite cements'. By definition blended cements are hydraulic binders in which a part of Portland cement is replaced by other hydraulic or non hydraulic materials. Their general behaviour is quite similar to that of Portland cement since they harden when mixed with water and form the same hydration products. The most common ingredients for blending with Portland cement clinkers are latent hydraulic component (blast furnace slag), or a pozzolanic component such as pozzolana, fly ash, rice husk ash, condensed silica fume, burnt clay or filler component such as lime stone and other waste materials (Hernandez et al., 1998; Massaza, 1979; Massaza, 1994; Mehta, 1987; Mehta, 1994; Narang, 1992; Rice Husk, 1979; Schmidt, 2004; Sersale, 1992; Singh et al., 2000; Uchikawa, 1986; Udachkin, 1992).

During hydration of Portland cement,  $\text{Ca}(\text{OH})_2$  is obtained as one of the hydration products, which in fact is responsible for deterioration of concrete.

But when certain pozzolanic materials containing amorphous silica is added during hydration of Portland cement, it reacts with lime giving additional amount of calcium silicate hydrate (C-S-H), the main cementing component. Thus the pozzolanic material reduces the amount of  $\text{Ca}(\text{OH})_2$  and increases the amount of C-S-H. Thus if a good quality pozzolanic material in suitable amounts, is added during the hydration of Portland cement, the cementing quality is enhanced. There is a continuous search for alternative supplementary materials, which may have hydraulic/pozzolanic properties. This is much more important in developing countries, where there is shortage of power and good quality raw materials. In an attempt to this, we found that ash obtained from bamboo leaf is amorphous in nature and has pozzolanic properties. The annual production of bamboos all over the world is about 20 million tonnes but about 10 million tonnes are produced in India, China and Japan (Vatsala, 2003). In this paper pozzolanic properties of bamboo leaf ash have been discussed.

## EXPERIMENTAL

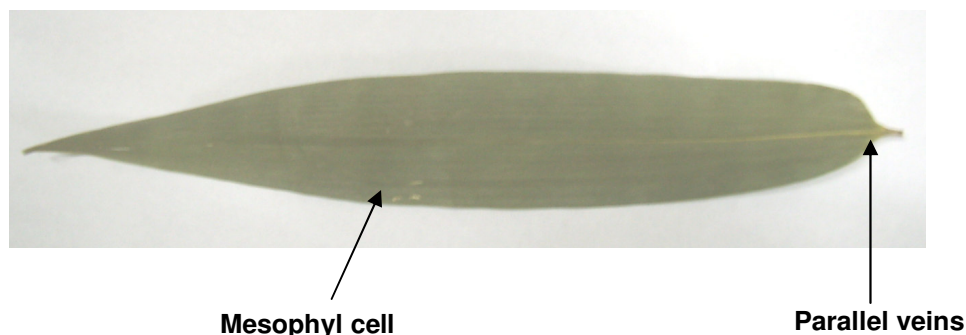
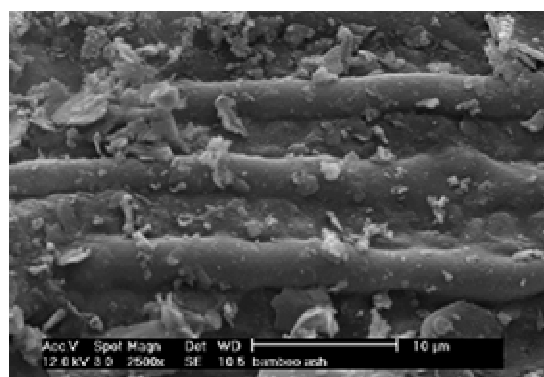
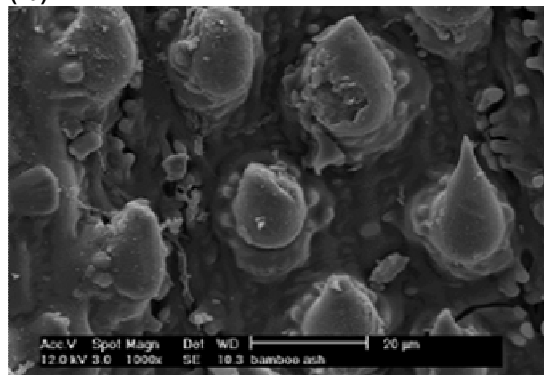
### Materials

Bamboo leaves were dried in sun, burnt in an open air and then heated in a muffle furnace at 600°C for 2 h to have bamboo leaf ash

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**Table 1.** Oxide compositions.

System	Composition (wt %)										
	SiO <sub>2</sub>	Al <sub>2</sub> O <sub>3</sub>	Fe <sub>2</sub> O <sub>3</sub>	CaO	MgO	K <sub>2</sub> O	Na <sub>2</sub> O	TiO <sub>2</sub>	SO <sub>3</sub>	IR	LOI
OPC	21.40	5.03	4.40	61.14	1.35	0.48	0.24	-	2.53	1.65	1.29
BLA	75.90	4.13	1.22	7.47	1.85	5.62	0.21	0.20	1.06	-	-

**Figure 1.** Photograph of Bamboo leaf**(2a)****(2b)****Figure 2.** SEM pictures of Bamboo leaf ash (a) Parallel veins (b) Mesophyll cell.

(BLA). Vikram premium Portland cement 53 (OPC) was used for hydration studies. Ca(OH)<sub>2</sub> (Qualigence) was used for studying pozzolanic reactions. The chemical composition of bamboo leaf ash and Portland cement are given in Table 1. The photograph of

bamboo leaf ash is shown in Figure 1. SEM pictures and powder X-ray diffraction pattern of bamboo leaf ash are given in Figures 2 and 3 respectively.

## Methods

**Reaction between calcium hydroxide and bamboo leaf ash:** 100g of Ca(OH)<sub>2</sub> and 100g of bamboo leaf ash were mixed thoroughly in polythene bags in presence of water in such a way that water/solid ratio was 1.0. The reactions were stopped at different intervals of time with isopropyl alcohol and ether. The samples were dried at 100°C and subjected to different analysis.

**X-ray diffraction studies:** X-ray diffraction studies of the reaction products obtained from reaction of calcium hydroxide and bamboo leaf ash, (prepared as mentioned above) were recorded with the help of a Philips PW1710 diffractograph using CuK<sub>α</sub> radiation.

**Differential scanning calorimetric (DSC) studies:** DSC studies of the reaction products obtained from reaction of calcium hydroxide and bamboo leaf ash at different intervals of time were recorded with a TG-DTG-DSC NETZSCH STA 409 instrument in the temperature range of 30 to 100°C in nitrogen atmosphere with a heating rate of 10°C/min using Al<sub>2</sub>O<sub>3</sub> as reference.

In order to determine the amount of calcium hydroxide reacted, a calibration curve was plotted. Ca(OH)<sub>2</sub> was mixed thoroughly with different amounts of Al<sub>2</sub>O<sub>3</sub> and DSC studies were made. A curve between heat of fusion of Ca(OH)<sub>2</sub> vs amount of Ca(OH)<sub>2</sub> was plotted, from which the amount of Ca(OH)<sub>2</sub> reacted, was calculated.

**Pozzolanic reactivity in suspension:** 100 mL of saturated Ca(OH)<sub>2</sub> solution was mixed with 20 g bamboo leaf ash and stirred magnetically. 10 mL of the solution was filtered at different intervals of time (1,2,3 and 4h) and was titrated against N/4 HCl using phenolphthalein indicator. From the titre values the amount of Ca(OH)<sub>2</sub> reacted with bamboo leaf ash was calculated. The experiment was performed at room temperature (~ 30°C) and 75°C.

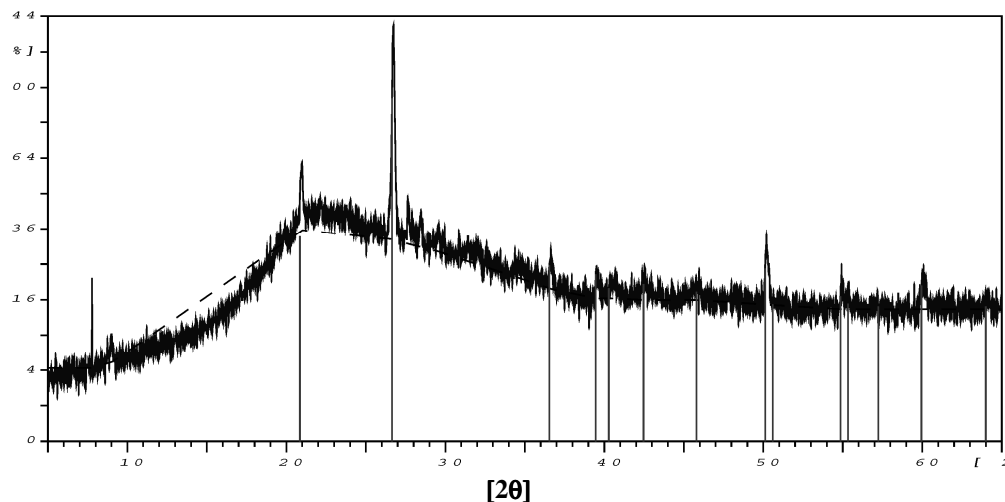


Figure 3. X-ray diffraction pattern of Bamboo leaf ash.

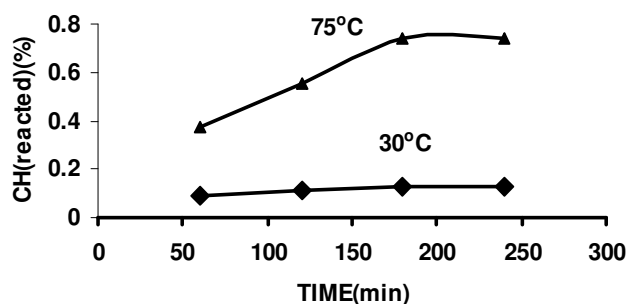


Figure 4. Amount of calcium hydroxide reacted at different time with BLA in suspension at different temperatures

**Preparation of hydrated samples of OPC and bamboo leaf ash blended cement:** 10 g of OPC or bamboo leaf ash blended cements (20 wt% bamboo leaf ash in Portland cement) were weighed separately in different polythene bags and mixed with 4 mL water so that w/s ratio became 0.4 and the air inside the bags was removed in order to avoid carbonations. The hydration reactions were allowed to continue at room temperature ( $\approx 30^\circ\text{C}$ ). The hydrations at different interval of time (1,3,7,14 and 28 days) were stopped with isopropyl alcohol and ether and the hydrated samples were heated at  $105^\circ\text{C}$  for 1 h. The dried samples were stored in polythene bags and kept in a desiccator.

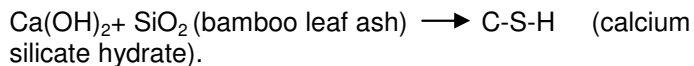
**Free lime determination:** One gram of hydrated cements (OPC/blended cement) was refluxed with 40 mL of isopropyl alcohol and acetoacetic ester mixture (20:3) for one hour. The solutions were filtered and titrated against 0.1N HCl using bromophenol blue as an indicator.

**Compressive strength measurements of Portland cement – Bamboo leaf ash composite cement:** Portland cement – bamboo leaf ash composite cements were mixed with sand in 1:3 ratio and then mixed with water (I S: 4031 part 4, 1988). The mortars were placed in steel moulds of  $70.6\text{ mm}^3$  dimension. These cubes were demoulded after one day and stored in water at  $27^\circ\text{C}$  at a relative humidity of 100%. These cubes were then taken out from water

prior to testing. The compressive strengths were determined at 3, 7 and 28 days as per I S: 4031 part 6, 1988 method.

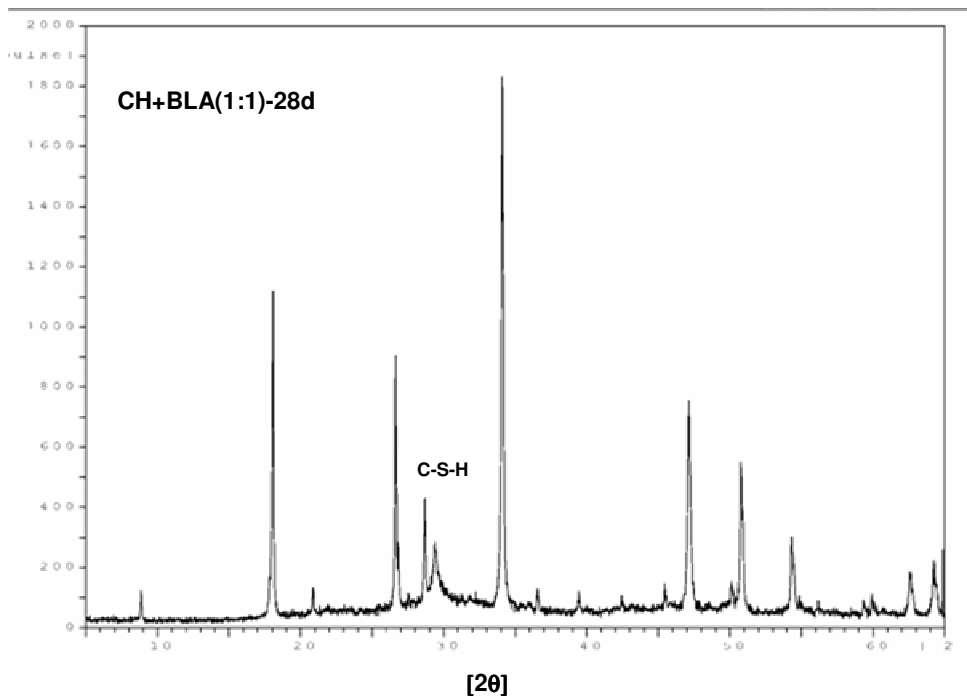
## RESULTS AND DISCUSSION

Bamboo leaf (Figure 1) consists of two parts (i) mesophyll cell and (ii) parallel veins. The SEM pictures of the ash (both parts) are shown in Figure 2. The X-ray diffraction pattern (Figure 3) of bamboo leaf ash heated at  $600^\circ\text{C}$  for 2 h shows that it is amorphous in nature. Since the ash contains amorphous  $\text{SiO}_2$ , it is expected to be a pozzolanic material and was allowed to react with calcium hydroxide in solution as well as in paste. In case of reaction in solution, bamboo leaf ash was mixed with saturated solution of calcium hydroxide and a definite volume of the solution (filtered) was titrated against standard HCl at different intervals of time. The amount of calcium hydroxide reacted with bamboo leaf ash was determined. The amount of reacted calcium hydroxide is plotted against time (Figure 4). The curve shows that as the time progressed, the amount of calcium hydroxide reacted was increased. The experiment was also performed at  $75^\circ\text{C}$  (Figure 4), where the reactivity was much higher than that at room temperature. The results suggest that bamboo leaf ash is a pozzolanic material and its pozzolanic activity increases with the increase in temperature. The pozzolanic reaction can be expressed as



The increase of rate of above reaction with temperature may be due to increase of dissociation of  $\text{Ca(OH)}_2$  in solution giving more  $\text{Ca}^{++}$  and  $\text{OH}^-$  ions. The larger number of ions react with amorphous silica at a faster rate.

Powder X-ray diffraction pattern of paste ( $\text{Ca(OH)}_2 +$  bamboo leaf ash) hydrated for 28 days is shown in Figure



**Figure 5.** X-ray diffraction pattern of CH+BLA hydrated for 28 days.

5. The figure shows unreacted calcium hydroxide and the formation of calcium silicate hydrate (C-S-H) phase. The diffraction pattern confirms that bamboo leaf ash is a pozzolanic material and during the course of reaction calcium silicate hydrate (C-S-H) is formed.

$\text{Ca}(\text{OH})_2$ +bamboo leaf ash pastes hydrated for different intervals of time were subjected to DSC studies. The DSC curves for the paste hydrated for 1, 28 and 90 days are shown in Figure 6. There are two endothermic peaks at about 100 and 475°C. The first peak is broad and is due to removal of water molecules and decomposition of C-S-H. Since at 1 day of hydration practically no C-S-H is formed, the peak is very broad but as the time progressed, the sharpness and peak area increased due to increased amount of C-S-H phase. The formation of C-S-H clearly indicates that bamboo leaf ash is a pozzolanic material and reacts with  $\text{Ca}(\text{OH})_2$ . The second peak at around 475 °C is due to decomposition of  $\text{Ca}(\text{OH})_2$ . From the figure one can see that at 1 day of hydration, very little reaction has taken place and the area for the decomposition of  $\text{Ca}(\text{OH})_2$  is maximum with maximum heat of decomposition. As the time progressed, the area of this peak and heat of decomposition decreased, showing that  $\text{Ca}(\text{OH})_2$  reacted with bamboo leaf ash and the extent of reaction increased with time.

The amount of calcium hydroxide reacting with bamboo leaf ash at different intervals of time was determined by DSC technique (Figure 7) using calibration curve. The curve shows that the amount of reacted calcium hydroxide increased with time indicating that the pozzolanic activity of bamboo leaf ash increased with time. However the

rate of increase of pozzolanic reactivity is much higher in the suspension. It appears that in suspension larger number of  $\text{Ca}^{++}$  and  $\text{OH}^-$  ions are available and as a result higher pozzolanic reaction.

In order to understand the role of bamboo leaf ash as a pozzolanic material during the hydration of Portland cement, the hydration studies of 20 wt% bamboo leaf ash blended Portland cement were made. Free lime values (Figure 8) were found to increase with time indicating an increase in hydration. In blended cement the values also increased with time but the values are much lower than that of OPC. This is due to reaction of  $\text{Ca}(\text{OH})_2$  obtained during hydration with amorphous silica of bamboo leaf ash.

The variation of compressive strength of cement mortar with time is shown in Figure 9. The compressive strength values increase with time but the values are lower in the case of blended cement. However at 28 days of hydration the compressive strength value of the blended cement is close to that of the control. At early time of hydration the lower values of compressive strength in the case of bamboo leaf ash blended cement may be due to lower pozzolanic reactivity. However the compressive strength value at 28 days of hydration is quite comparable to that of control, which may be due to additional amount of calcium silicate hydrate formed as a result of pozzolanic reaction.

Structure developments leading to strength in hydrating cement pastes are due to formation of different hydration products which are both crystalline and amorphous in nature and also due to contact of different surfaces. The

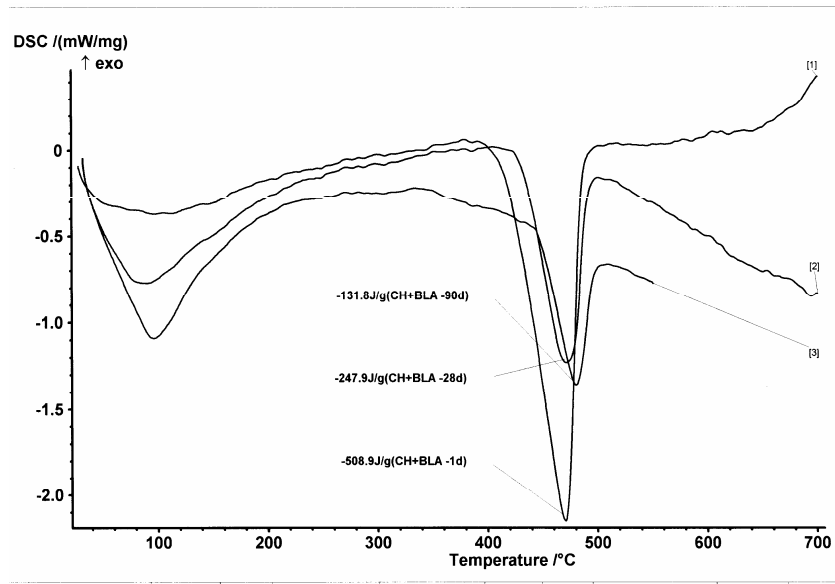


Figure 6. DSC curves of CH+BLA hydrated pastes.

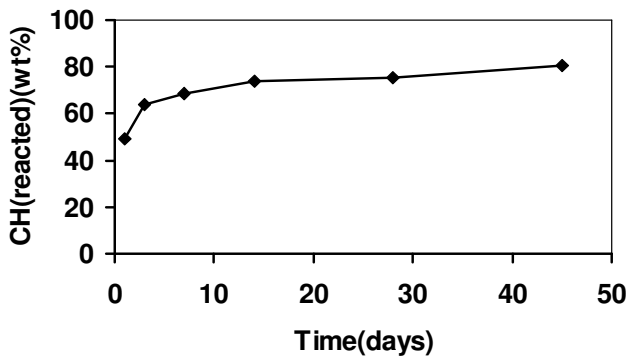


Figure 7. Amount of calcium hydroxide reacted with BLA in a paste at different time as determined by DSC technique

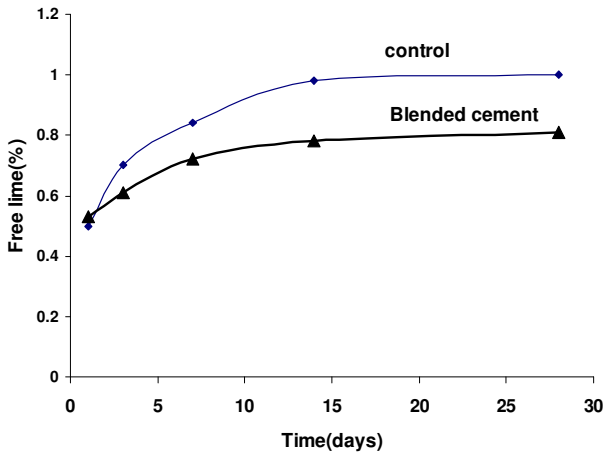


Figure 8. Variation of free lime with hydration time

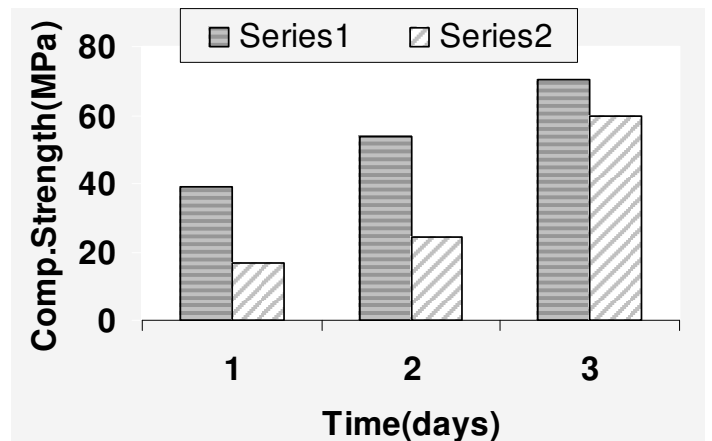


Figure 9. Variation of compressive strength with time. Series1- OPC, Series2- Blended cement

increase in cohesion is then due to increase in facing surfaces. According to Powers (Powers, 1958) physical forces (between surfaces) are responsible for the development of strength. However chemical forces contribute little. In the presence of bamboo leaf ash, quantities of hydration products change and as a result, surface contacts also change. Thus it appears that in the presence of bamboo leaf ash, surface forces change leading to microstructural and strength changes. Nachbaur et al (Nachbaur et al., 2001) have also shown that physical forces between particles are responsible for mechanical properties of the cement pastes from mixing to setting and even later.

## Conclusions

The experimental results show that bamboo leaf ash is a good pozzolanic material which reacts with calcium hydroxide forming calcium silicate hydrate. The pozzolanic activity of bamboo leaf ash increases with increase of time and temperature.

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