Journal of Archaeological Science 37 (2010) 1339–1347

Contents lists available at ScienceDirect

Journal of Archaeological Science

journal homepage: http://www.elsevier.com/locate/jas

The use of volcanic materials for the manufacture of pozzolanic plasters in the Maya lowlands: a preliminary report

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ARTICLE INFO

Article history:
Received 6 October 2009
Received in revised form
22 December 2009
Accepted 23 December 2009

Keywords:
Maya
Lime plasters
Pozzolanic plasters
Volcanic glass
Calakmul
Lamanai

ABSTRACT

This paper focuses on the description and characterization of the nature of isotropic and silicon-rich particles observed in plaster samples from the Maya archaeological sites of Calakmul and Lamanai. Based on the composition and morphology of these particles, it is proposed that volcanic ash and glass were incorporated into some of the ancient Maya plasters in order to produce hydraulic reactions. From a technological point of view, the intent on the part of the Maya to produce such reactions is relevant because it would reflect resource planning and complex knowledge of materials. Results also confirm previous reports of the presence of volcanic ash layers at the site of Calakmul and suggest that there were considerable ash falls in the Maya lowlands during Prehispanic times which may have made the material available in many areas of the Central Lowlands. If, as we propose here, volcanic ash was employed in lowland Maya plasters, then the use of volcanic materials by the lowland Maya was more intensive than has been recognized. Our paper also reviews the literature about the use of volcanic materials in lowland Maya materials and suggests future lines of research.

1. Lime production and the manufacture of lime plasters

Lime plasters are mixtures of slaked lime, aggregates and other materials that are employed in masonry architecture. Non-hydraulic limes are produced when limestone or another calcium carbonate-rich material is burnt at temperatures around 900 °C, and the compound is transformed into calcium oxide, CaO. This material is then slaked with water or moist air, forming a white powder or paste depending on the amount of water, and transforms into calcium hydroxide, $Ca(OH)_2$. The slaked product is sometimes stored for several months to promote hydration and to improve plasticity and other working properties of the lime. During setting and following exposure to air, $Ca(OH)_2$ reacts with carbon dioxide to form calcium carbonate, $CaCO_3$, a process that can take several months or years to complete (Boynton, 1980).

Non-hydraulic limes harden solely by drying and carbonation, in contrast with hydraulic limes and Portland cement which set under water by the formation of a variety of compounds that incorporate water in their structure. Hydraulic limes can be obtained by burning limestones with clay impurities, or by deliberately mixing $CaCO_3$ with clays or clay-like materials containing silica and alumina before calcination. In this case, clay minerals react with $CaCO_3$ during the calcination process to form silicates and aluminates that are then hydrated during the slaking stage to form silicate and aluminate hydrates (Boynton, 1980). Similar limes to hydraulic limes, known as pozzolanic limes, can be produced by mixing pozzolanas with slaked lime, which results in the formation of a range of hydraulic compounds, including calcium silicate and aluminate hydrates. The most common type of pozzolanas for the manufacture of pozzolanic plasters in antiquity was reactive silica in the form of volcanic ash, as well as ceramic powder. The pozzolanic activity of these aggregates has been studied by Moropolou et al. (2004), who showed that volcanic ash (Earth of Milos) presents a greater pozzolanic reaction in comparison to ceramic powder. At present there is a range of materials that can be used as pozzolanas, such as fly ash or condensed silica fume (King, 2000).

Broadly speaking, hydraulic compounds confer characteristics that may be desirable on the plasters, such as a higher compressive strength in comparison with non-hydraulic limes. Hydraulic compounds also result in a less soluble material with the ability to set under water, although the specific properties also depend on the type of pozzolanic aggregate employed (Gibbons, 2003), the milling degree of the raw materials, which considerably increases their reactivity (Miriello et al., In press), and the fine grain size of the raw materials, which results in a lower porosity (Farci et al., 2005).

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0305-4403/$ – see front matter
doi:10.1016/j.jas.2009.12.038

According to Dandrau (2000), hydraulic plasters were used by the Minoans, although the Romans are generally recognized to be the first to use pozzolanic aggregates systematically by mixing slaked lime with volcanic ash from the town Pozzuoli, a source of volcanic ash from Vesuvius (Siddall, 2000). The town gave its name to the term "pozzolanic aggregates", which is applied to materials that have reactive properties when mixed with lime. The pozzolanic Roman plasters were known as *opus caementicium* and were described by Vitruvius Pollio (1999, book II [ca. 25 B.C.]) in his *Ten Books of Architecture*.

Ancient cultures employed hydraulic and pozzolanic plasters for a variety of purposes. The hydraulic-setting property was exploited by the Romans for construction of harbors by pouring the mixtures directly under water (Oleson et al., 2004). The Romans also employed pozzolanic plasters for lining cisterns, because they are more durable than non-hydraulic plasters and therefore retain water more efficiently (Rizzo et al., 2008).

In contrast to reliance on historical evidence, the identification of volcanic ash in archaeological and historical plasters is a difficult task, owing to the fact that the reaction of the lime with silica frequently leaves behind no identifiable minerals that can be observed in the hardened plaster under the petrographic microscope.

1.1. The production of lime and lime plasters in the Maya area

Lime was produced and used extensively in Mesoamerica for building purposes as early as the Middle Preclassic period (1000–400 B.C.), although its production may have long preceded its use in architecture. The earliest evidence of the use of lime in Maya architecture is found in the Central Lowlands during the Middle Preclassic Period at the sites of Cuello, Belize (Andrews and Hammond, 1990; Gerhardt, 1988; Hammond and Gerhardt, 1990), Nakbé, Guatemala (Hansen et al., 1995, 1997), and Calakmul, Mexico (García-Solís et al., 2006). The use of lime continued throughout the Prehispanic period across the Maya area, and ancient techniques have been passed down generations to the present day. It is also well known that lime was used for tobacco and maize soaking before the arrival of the Europeans, as it is still used today. Lime increases the hallucinogenic properties of tobacco, and in maize treatment it softens the grain, allowing removal of the pericarp, and also enhances nutritional properties (Katz et al., 1974).

Ethnographic research describes sophisticated lime-production techniques in contemporary Maya communities, especially regarding the construction of open pyres for the burning of limestone, a technology that is often embedded in a ritual context (Schreiner, 2002; Russell and Dahlin, 2007). This is a significant technological difference in comparison to Old World lime production, where enclosed kilns have been used since early times. Many of the contemporary Maya techniques are likely to have origins which can be traced back to Prehispanic times but the archaeological evidence of lime production in the Maya area is remarkably scarce.

Despite the fact that lime plasters were extensively used in Maya architecture, plasters have often been overlooked in archaeological research, although there has been some recent interest in the characterization of raw materials and other technological aspects (Brown, 1986a,b,c,d,e; García-Solís et al., 2006; Goodall et al., 2007; Hansen, 2005; Hansen et al., 1995, 1997, 2001; Littman, 1959a,b; 1960a,b; 1962, 1966, 1967, 1990; Magaloni et al., 1995; Villegas et al., 1995; Villaseñor and Aimers, 2009). Hydraulic or pozzolanic plasters in the Maya area have not, however, been identified and characterized by researchers.

2. The use of volcanic materials in the Maya Lowlands

Many authors (Ford and Glicken, 1987; Jones, 1986; Kidder, 1937; Rands and Bishop, 1980: 23; Shepard, 1939, 1956, 1964; Simmons and Brem, 1979) have reported the presence of volcanic ash in lowland Maya ceramics. In addition, Simmons and Brem (1979: 80) have assembled information on the chronological distribution of occurrences of volcanic ash in lowland ceramics (Fig. 1).

Although Isphording and Wilson (1974) claim that the volcanic ash identified by Shepard (1939, 1956, 1964) was palygorskite, reexamination of the material has confirmed Shepard's identification of volcanics in lowlands ceramics (Simmons and Brem, 1979). The presence of volcanic material in lowland pottery has prompted some debate and speculation regarding provenance, which has been proposed to be the Guatemalan Highlands, from which the material arrived in the lowlands in exchange for salt from the Northern Lowlands (Simmons and Brem, 1979).

In contrast, Ford and Rose (1995) argue that, in order to account for the quantity of volcanic ash found in lowland Maya pottery during Classic times, there must have been local sources of procurement. The authors propose that such sources result from a period of active volcanism that lasted several centuries and produced numerous ash falls that covered the Maya lowlands. Espíndola et al. (2000) and Peralta (2004) believe that the El Chichón volcano in Chiapas erupted frequently in Prehispanic times, covering

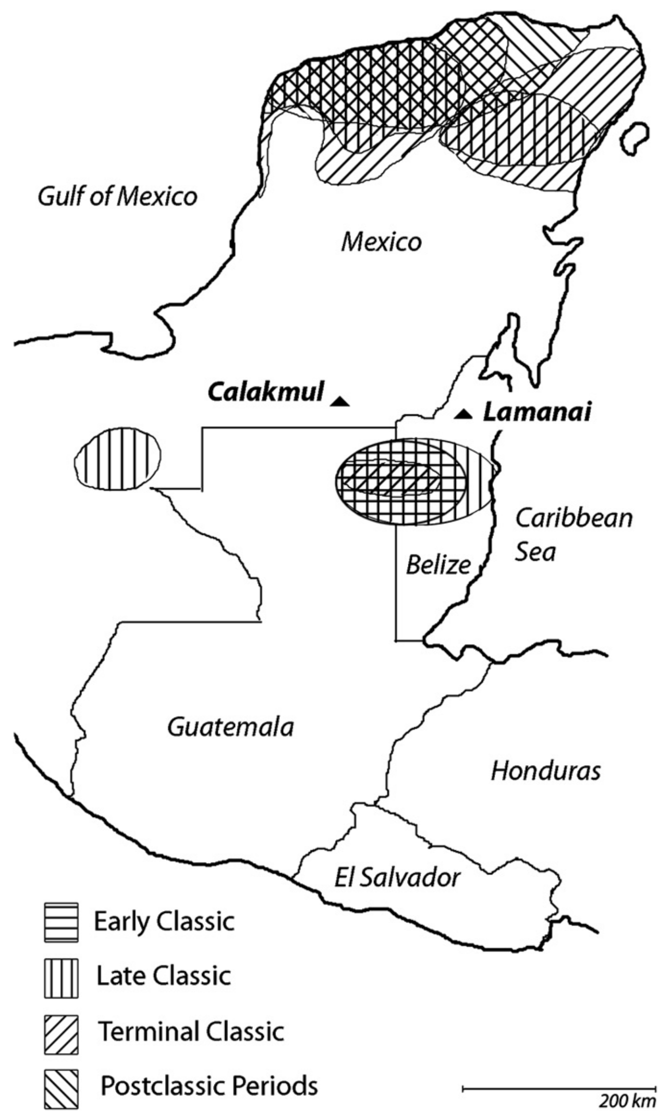

Fig. 1. Distribution of ash-tempered ceramics in the Maya area. Adapted from Simmons and Brem (1979:80).

areas of the Western Maya Lowlands, as happened in 1982, when ash falls reached Belize as well as the states of Veracruz, Tabasco, Oaxaca and Campeche, a radius of 200 km.

Graham (1987) has also suggests the availability of local resources and notes that there are much older volcanic deposits that occur in Belize in the form of welded tuffs and ashes south of the Pine Ridge Batholith and throughout the outcrop of the Bladen Volcanic Member of the Santa Rosa Group (Bateson and Hall, 1977; Hall and Bateson, 1972, cited by Graham, 1987; Abramiuk and Meurer, 2006), as well as pumice fragments which wash up in mangrove swamps and onto beaches along the Belize coast (Graham, 1994:312). Many of these volcanics and volcaniclastics were utilized by the ancient Maya, as in the case of the volcanic rocks employed for the production of manos and metates (Abramiuk and Meurer, 2006).

Volcanic ash deposits have also been found in core samples from bajos (swampy areas) in the Petén (Gunn et al., 2002); their presence demonstrates that tephra from volcanic eruptions, either from Chiapanecan or Guatemalan volcanoes, reached the central area of the lowlands.

In addition to the use of volcanic materials for the production of lowland Maya ceramics, Barba et al. (2008) have reported the presence of volcanic glass shards in plasters from Teotihuacan, Mexico, although the authors report that there is no hydraulic reaction between the lime and the volcanic ash. Magaloni (1995), in her study of Teotihuacan plasters, also shows photomicrographs with visible glass shards. In the same way, Hansen et al. (1997) report that Late Classic plasters from Nakbé, Guatemala, exhibit considerable hardness that may be due to the presence of hydraulic compounds. While these three examples suggest that Mesoamerican peoples had discovered the benefits of mixing lime with reactive volcanic materials, many questions remain regarding the locations of volcanic deposits exploited, the periods in which reactive volcanic materials were used in Maya plasters, and the specific hydraulic compounds that are found in ancient Maya plasters.

3. Analytical procedures

We analyzed 21 plaster samples from Calakmul and 36 samples from Lamanai. All samples were taken from either elite residences or civic-ceremonial architecture located in the core of the sites. The analytical techniques employed were petrography and scanning electron microscopy with energy dispersive spectrometry (SEM-EDS) with the aim of understanding the nature of the raw materials and their technological implications. Petrography was employed to characterize the mineralogy of the samples and observe their micromorphological characteristics, whereas SEM-EDS was employed in order to obtain a semi-quantitative elemental composition of the different components previously observed in the samples with the petrographic microscope. Samples included material from the Late Middle Preclassic period (ca. 400 B.C.) to the Terminal Classic period (ca. A.D. 900) in the case of Calakmul, and from the Late Preclassic (ca. 100 B.C.) to the Early Spanish Colonial period (ca. A.D. 1600) in the case of Lamanai. The samples, which measured approximately 2 cm^2 were taken from floors and wall renders with hammer and chisel. From the samples analyzed, we present the photomicrographs and semi-quantitative composition of 6 samples from Calakmul and 4 samples from Lamanai in which we identified the presence of volcanic materials and/or evidence of hydraulic compounds (Tables 1 and 2).

Table 1
Samples reported in this paper showing the location and chronology of the samples, as well as the analytical techniques carried out in each of them.

Samples information				Si-rich and isotropic materials						Other features		Analytical techniques	
Sample code	Site	Description	Location/Type of architecture	Time period	Isotropic layers	Isotropic materials in plasters	Devitrified glass	Silica gel	Acicular crystals	Reaction rims around isotropic materials	Mottled appearance of the matrix suggesting hydraulicity	Petrography	SEM/EDS
Ca6	Calakmul	Wall render	Substructure II c 1/Civic-ceremonial	Late Middle Preclassic		*			*			*	*
Ca8	Calakmul	Stucco sculpture	Substructure II c 2/Civic-ceremonial	Late Middle Preclassic.		*	*		*			*	*
Ca11	Calakmul	Wall render	Substructure IId/Civic-ceremonial	Middle Preclassic		*			*	*	*	*	*
Ca31	Calakmul	Floor	Pit in front of structure VII/Civic-ceremonial	Early Classic (?)	*							*	*
Ca16	Calakmul	Wall render	2nd body, Str. I-1/Civic-ceremonial	Late Classic		*	*		*			*	*
Ca18	Calakmul	Floor	Northeast structure, St. GN-1/Civic-ceremonial	Late Classic		*	*		*			*	
La9	Lamanai	Floor	N10-15/Elite residence	Late/Terminal Classic		*					*	*	
La49	Lamanai	Floor	Str. N12-11 (YDLI)/Civic-ceremonial	Late Postclassic		*					*	*	*
La36b	Lamanai	Wall render?	Pit west to Str. N12-11 (YDLI)/Civic-ceremonial	Late Postclassic?		*	*	*			*	*	*
La20	Lamanai	Joining mortar	Str. N12-13 (YDLII)/Civic-ceremonial	Spanish Colonial			*				*	*	*

Table 2
Normalized semi-quantitative elemental composition of glass shards and other isotropic materials as measured by EDS analysis. Ca: Calakmul; La: Lamanai; JM: joining mortar; W.R: wall render; F: floor; Sculp: sculpture; L.M. Prec: Late Middle Preclassic; M.Prec: Middle Preclassic; E.Clas: Early Classic; Late Postclassic; S.Col: Spanish Colonial; n.d: not determined.

Sample	Sample description	Area description	SiO_2	TiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	SO_3	Br	Totals
Ca6	W.R., L.M. Prec.	Globular particles with acicular crystals.	99.3	n.d.	n.d.	n.d.	n.d.	0.7	n.d.	n.d.	n.d.	n.d.	100
Ca8	Sculp. L.M. Prec.	Acicular crystals.	11.2	n.d.	n.d.	n.d.	n.d.	88.8	n.d.	n.d.	n.d.	n.d.	100
Ca11	W.R., M.Prec.	Reaction rims (hydraulic reactions)	51.7	n.d.	n.d.	n.d.	0.4	46.2	n.d.	n.d.	0.8	1.0	100
Ca31	F, E.Clas (?).	Isotropic layer (volcanic ash) (Fig. 5)	93.1	0.2	2.3	0.6	0.4	2.6	n.d.	0.1	0.6	n.d.	100
Ca16	WR, L.Clas.	Devitrified glass	73.1	n.d.	11.8	2.6	9.3	1.7	n.d.	1.0	0.5	n.d.	100
La49	F, L.Post.	Glass around quartz (Fig. 3).	71.2	n.d.	17.7	n.d.	0.4	9.1	0.3	1.4	n.d.	n.d.	100
La36b	WR, SC (?)	Rounded isotropic particle	52	1.3	39.1	1.6	0.8	2.3	n.d.	0.3	2.6	n.d.	100
La36b	WR, SC (?)	Devitrified glass shard.	54.8	0.7	27.9	12.9	1.3	1.6	n.d.	0.7	n.d.	n.d.	100
La20	JM, SCol.	Devitrified glass shard (Fig. 2).	55.9	1.1	34.7	3.1	1.4	2.4	0.3	1.0	n.d.	n.d.	100

Thin sections for petrography were prepared according to standard procedures and petrographic observations were carried out with a CMLP petrographic microscope in magnifications ranging between 40 and 630×; pictures were taken with a digital camera attached to the microscope.

For the SEM analysis, a thin-film window Hitachi S-570 was used to obtain compositional images of polished thin sections with the backscattered electron mode. EDS semi-quantitative analyses of the different components were carried out and the reported data are the average of three different measurements of the same area under study, with oxygen combined by stoichiometry and normalized to 100%. In the case of the layer of volcanic ash (sample Ca31) the three different measurements are presented (Table 3), given the relevance for associating it with specific eruptions. Acceleration voltage was 15 and 20 kv with a working distance of 10 mm.

4. Results

Petrographic analysis revealed a range of sample components. A carbonate matrix was present in which carbonate aggregates predominated; grains of monocrystalline and polycrystalline quartz were also observed in about half of the samples. In addition, observation revealed the presence of isotropic materials–materials not affected by polarized light– in 14 of the 21 samples from Calakmul and 17 of the 36 samples from Lamanai. Some of the isotropic inclusions were analyzed with SEM/EDS equipment and proved to have silicon dioxide (SiO_2) as the major component, followed by aluminium oxide (Al_2O_3) (see Table 2).

In the case of Lamanai, some of the inclusions consisted of sickle-shaped fragments (Samples La20, La36b: Fig. 2), often with an earthy appearance, which proved to have high contents of SiO_2 with significant amounts of Al_2O_3 and iron oxide (FeO) (see Table 2 and Fig. 2). In other cases, rounded fragments of glass were observed (Sample La36b), as well as fragments of quartz crystals embedded in glass, the composition of which corresponds to dacitec rocks (Le Maitre, 1989) (Sample La49; see Fig. 3 and Table 2).

In contrast, in the case of Calakmul the isotropic materials were observed as masses without defined shapes (Sample Ca18; Fig. 4). In addition, a thin isotropic layer of around 200 μm thick with vesicles throughout was observed atop a plaster from a floor dating to the Early Classic period from Calakmul (Sample Ca31; See Figs. 5 and 6). On occasion, visible reaction rims were also present around isotropic materials (Sample Ca11), together with areas with a mottled appearance and low optical activity of the lime binder.

Acicular crystals were also observed with the petrographic microscope and the SEM in many samples from Calakmul, mainly from the Preclassic periods: Ca6, Ca8, Ca11, Ca16 and Ca18 (see Fig. 7). In the case of sample Ca16, from the Late Classic period, acicular crystals were seen in apparent association with silicon-rich, partly isotropic particles. Acicular crystals in sample Ca8, from the Late Middle Preclassic period, were analyzed with the SEM and showed a composition of 77 % CaO and 18 % SiO_2 although analytical totals before normalization were very low, probably owing to the shape and location of the crystals.

5. Discussion

Examination of archaeological plasters from Calakmul and Lamanai revealed numerous samples with isotropic materials rich in SiO_2, acicular crystals and reaction rims. From the samples analyzed, volcanic materials and hydraulic reactions were identified in six samples from Calakmul and four samples from Lamanai (see Table 1). Based on the composition, shape and optical properties, the isotropic materials appear to be composed of volcanic ash and fragments of solidified volcanic ash or dust (Figs. 2–8).

Volcanic ash and glass are formed during eruptions when magma is cooled down too quickly to allow any crystalline structure to develop, and forms a glass or vitrophyre. Volcanic ash is most commonly of rhyolitic composition, i.e. more than 70 % SiO_2 (Tarbuck and Lutgens, 2002), and it is precisely the chemistry and non-crystallinity of these materials that make them suitable to be used for the production of pozzolanic plasters.

5.1. Lamanai plasters

As can be seen in Table 2, the samples that show isotropic particles rich in silicon date to the Late Postclassic and Early Spanish Colonial periods. A frequently observed mineral in Late Postclassic and Spanish Colonial samples from Lamanai is quartz, which is often embedded in silicon dioxide-rich glass. This is a characteristic of acid volcanic rocks, in which free quartz is formed alongside glass. Analysis of the glass surrounding quartz crystals in sample La49 with the SEM/EDS revealed a composition corresponding to that of volcanic glass (see Table 2 and Fig. 3).

Table 3
Normalized semi-quantitative elemental composition of isotropic layer (microtephra) in sample Ca31 as measured by EDS analysis (Figs. 5 and 6). F: floor; E.Clas: Early Classic; n.d: not determined.

Sample	Sample description	Area description	SiO_2	TiO_2	Al_2O_3	FeO	MgO	CaO	Na_2O	K_2O	SO_3	SnO_2	HgO	Totals
Ca31	F, E.Clas (?).	Isotropic layer (volcanic ash) (Fig. 5)	92.8	0.2	2.3	0.6	0.4	2.6	n.d.	n.d.	n.d.	n.d.	1.0	100
			62.5	0.3	4.4	1.2	1.0	16.1	n.d.	0.5	14.1	n.d.	n.d.	100
			95.0	n.d.	1.7	0.4	0.3	0.7	0.2	n.d.	n.d.	0.3	1.5	100

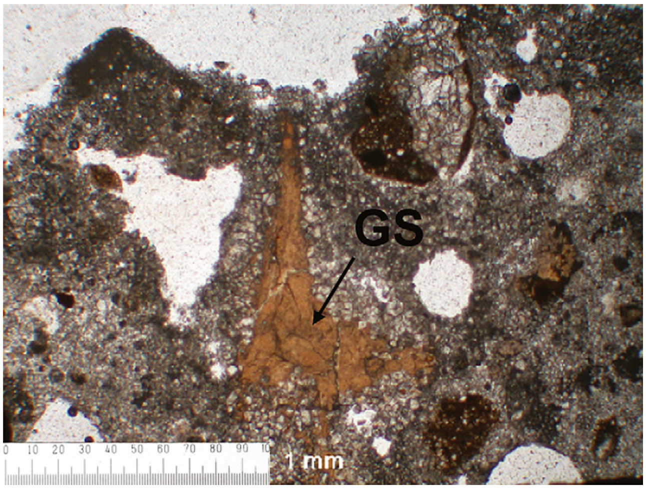

Fig. 2. Sample La20, Spanish Colonial period, Lamanai. Devitrified glass shard (GS) (see Table 2 for composition). Transmitted plane polarized light. Scale bar: 1 mm (10 units represent 100 μm).

The particles with earthy appearance that occur in some of Lamanai samples (see Fig. 2) have the characteristic sickle shape of volcanic glass shards. The yellow colour in the particles indicates chemical weathering, which is caused by the reaction of the glass with low-temperature waters to form clay minerals (Vaniman, 2006:13).

The closest volcanic deposits to the site of Lamanai are found in the Bladen Volcanic Member, which dates to the Late Palaeozoic and is about 285 million years old, (Donnelly et al., 1990). These deposits are contemporaneous with the volcanic materials of the northern Guatemalan Highlands but considerably older than the Quaternary volcanic activity of the southern Highlands, the ashes from which may have reached the Maya lowlands during ancient Maya times (Ford and Rose, 1995). Abramiuk and Meurer (2006) have demonstrated that the Bladen volcanics were exploited by the Bladen Maya communities in Precolumbian times for the manufacture of manos and metates. It is also worth noting that ash-tempered ceramics dating from the Early, Late and Terminal Classic periods have been found in northern and central Belize (Simmons and Brem, 1979:80, Fig. 1). Considering the alteration of the observed glass shards into clays, the proximity of the Maya Mountains and the proven exploitation of volcanic materials in the area, the Bladen Volcanic Range emerges as a likely source for the weathered volcanic glass fragments observed in the plasters from Lamanai.

In addition to the presence of altered glass fragments, Late Postclassic and Early Spanish Colonial samples from Lamanai show some areas with apparent hydraulicity, with the characteristic mottled appearance and low optical activity of the lime binder. Some of these plasters also exhibit rounded isotropic materials rich in silica, which are likely to correspond to alkali-silica gels (see Sample La36b; Table 2), and which are often seen in hydraulic limes (St. John et al., 2003), although they can also constitute eroded fragments of glass shards. All of the foregoing characteristics seem to suggest that the Maya exploited reactive siliceous deposits during the Late Postclassic and Early Spanish Colonial periods in order to produce plasters with some degree of hydraulicity. Although more research is needed, it is evident that during these periods plasters were mixed with materials that had not been employed before and are likely to be non-local in origin. However, because the sampling of earlier plasters was limited, the possibility remains that pozzolanic plasters were produced in earlier periods, which is an aspect that needs further investigation.

If volcanic aggregates were indeed first used in plaster production at Lamanai in the Late Postclassic, then their appearance at this relatively late point needs to be explained. Community-wide participation in trade and commercial activities seems to characterize the Postclassic period at Lamanai (Graham, 2004), and it is therefore tempting to suggest expansion of trade networks as an explanation for the introduction of volcanic aggregates. On the other hand, ground stone from southern Belize was reaching Lamanai from Preclassic times onward and it therefore seems unlikely that trade patterns new to the Late Postclassic would have been responsible for access to volcanic aggregates. A further possibility is that plaster-workers at Lamanai first became aware of the advantages of volcanic aggregates in the Late Postclassic.

In the case of the historic-period plasters of Lamanai, the incorporation of volcanic materials is most likely to have been the result of continuity in Maya technological practices from the Late Postclassic period. In Europe, although pozzolanic plasters and their description in Classical treatises such as Vitruvius Pollio (1999, book 2[ca. 25 B.C.]) and Palladio's (1998, book 1 [A.D. 1570]) were known, the systematic use of hydraulic and pozzolanic limes in Europe did not restart until the 18th century, reason why the use of volcanic glass was probably not a Spanish introduction.

It has to be noted, however, that most of the Late Postclassic and Spanish Colonial samples were wall renders, in contrast with the rest of the samples of previous periods, which were mainly floors. We therefore do not know if the incorporation of volcanic materials was a general practice in all Late Postclassic and Spanish Colonial plasters, or if earlier wall renders might have incorporated volcanic materials.

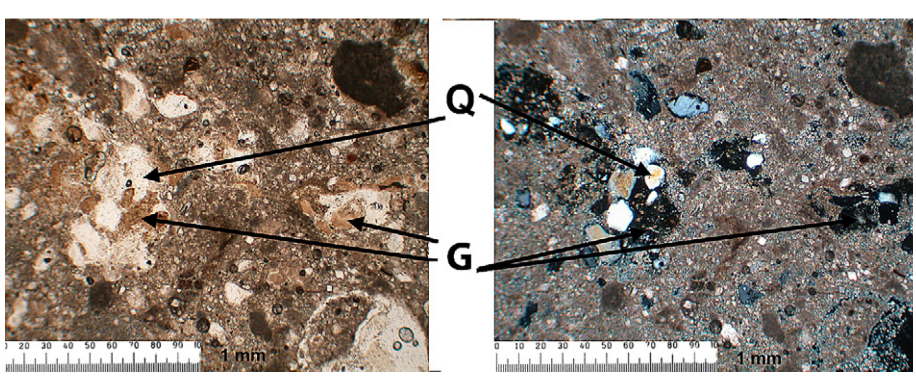

Fig. 3. Sample La49, Late Postclassic period, Lamanai. Fragments of acid volcanic rocks composed of quartz (Q) and glass (G) (see Table 2 for composition). Left: Plane polarized light, right: crossed polarized light. Scale bar: 1 mm (10 units represent 100 μm).

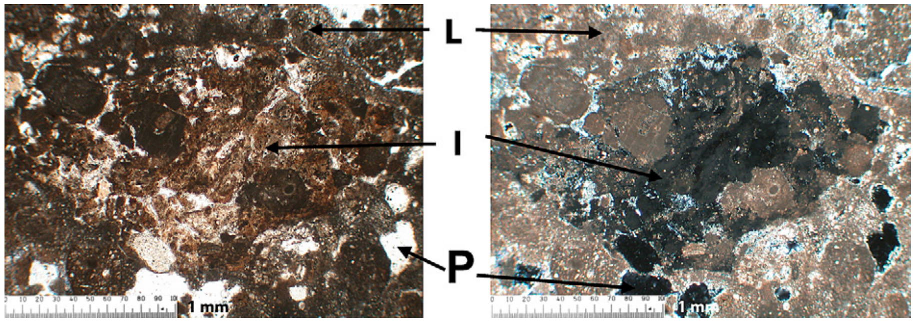

Fig. 4. Sample Ca18, Late Classic period, Calakmul. Lime matrix (L), Isotropic particle (fragment of solidified volcanic ash) with visible pipe vesicles (I), and pores (P). Left image: plane polarized light. Right image: crossed polarized light. Scale bars: 1 mm (10 units represent 100 μm).

5.2. Calakmul plasters

In the case of Calakmul, acicular crystals were frequently seen in Preclassic samples (see Fig. 7). As Charola and Henriques (1999:6) and Goldsworthy and Min (2009) describe, crystalline habits of this sort are often the most clearly seen evidence of hydraulic components and frequently grow in calcium silicate hydrate (C–S–H). Although the presence of acicular crystals is not conclusive evidence of hydraulic or pozzolanic plasters, because these habits are also found in carbonate sediments and rocks (Scholle and Ulmer-Scholle, 2003:337), acicular crystals in Sample Ca8, as mentioned above, were analyzed with the EDS and proved to have high silicon content, which does suggest the presence of hydraulic compounds. Moreover, in the case of Late Classic samples, acicular crystals were seen in association with yellow glass (Ca16), which suggests that the crystals were formed as reaction products of the lime and glass.

In a similar way, the samples with isotropic particles high in SiO_2 come from the Late Classic period. In contrast with the characteristics of the Lamanai glass shards, the isotropic materials from Calakmul appear as masses without defined shapes. The masses comprise minute particles that are not possible to recognize individually under the petrographic microscope, and which all evidence suggests must represent fragments of solidified volcanic ash or clusters of submicroscopic particles of volcanic dust, such as those reported by Ford and Rose (1995) in the case of ceramics from the Tikal Yaxha region. The use of volcanic ash in Late Classic plasters at Calakmul is paralleled at this time by an increase in the production of ash-tempered ceramics (Domínguez et al., 2004).

In the case of Sample Ca31 (Fig. 5), the isotropic layer is primarily composed of SiO_2, with some contents of Al_2O_3 and CaO, which corresponds well with the nature of volcanic ash, and it is most likely to be a layer of long-distance microtephra (solidified volcanic ash) deposited over a plaster floor. The content in sulphur, however, proved to be highly variable, and may be related to the embedding and adhesive resins employed for thin sectioning. The presence of gypsum ($CaSO_4 \cdot 2H_2O$) can be ruled out because no gypsum was observed in the isotropic layer by means of petrography. On the other hand, the extremely high contents in SiO_2 may be a distortion of the analysis, which is known to be caused by effects of the beam size, the small size of the glass shards and the thinness of their walls (Hunt and Hill, 2001).

The microstructure of this layer, consisting of many small vesicles (Fig. 6), as well as the optical properties, consisting on isotropy under cross polars and a brownish-yellow colour under plane polarized light with the absence of phenocrystals distinctively larger crystals than the groundmass (Fig. 5), corresponds well with the characteristics of distal microtephra, that is to say, long-distance wind-transported pyroclastic materials (see Heiken, 1972).

It is worth noting in this respect that a layer of volcanic ash had previously been reported by Gunn et al. (2002) in the case of a seasonally-inundated swamp or *bajo*. Nevertheless, the layer in Sample Ca31 constitutes, to the best of our knowledge, the only reported occurrence of volcanic ash deposited over anthropogenic material in the Maya lowlands.

It is possible that microscopic tephra horizons were deposited during a period of active volcanism of a Guatemalan or Chiapanecan volcano. Although it may appear that Calakmul is too far from the volcanic area to be reached by ash falls, it is well known that microtephra can be dispersed over thousands of kilometres, the distance of dispersal depending on the height to which pyroclastic material is thrown into the air, as well as other factors such as wind and rain (Hall, 1996:48). One possible source for the tephra could be the eruption of the Ilopango volcano, in central El Salvador, which occurred early in the first millennium A.D. and produced a long distance ash spread (Steen McIntyre, 1981:360 cited by Jack, 2005). Other possible sources for this tephra could be the Chichón, Cerro Quemado and the Amatitlán volcanoes, which have dated eruptions during the Classic period (Ford and Rose, 1995).

Comparison of the isotropic layer of sample Ca31 and the isotropic inclusion in sample Ca18 (Figs. 4 and 5), makes it clear that

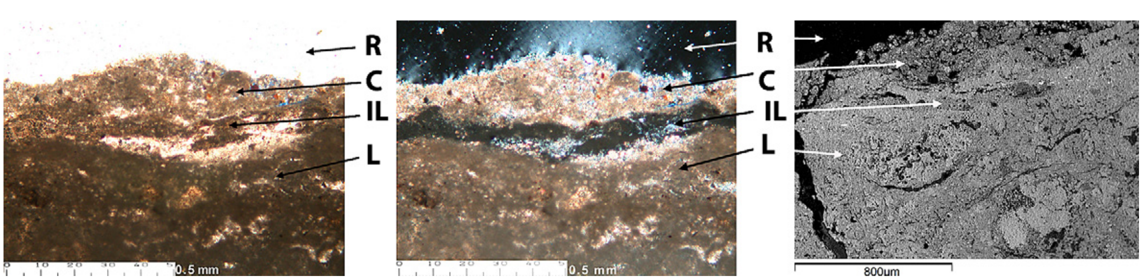

Fig. 5. Sample Ca31, Early Classic period, Calakmul. Embedding resin (R), carbonate fill deposited over the floor (C), isotropic layer rich in silicon (layer of microtephra, see Table 3 for composition) (IL), lime plaster floor (L). Left image: transmitted plane polarized light. Central image: plane polarized light, scale bars: 500 μm (10 units represent 100 μm). Right image: backscattered electron image, scale bar: 800 μm.

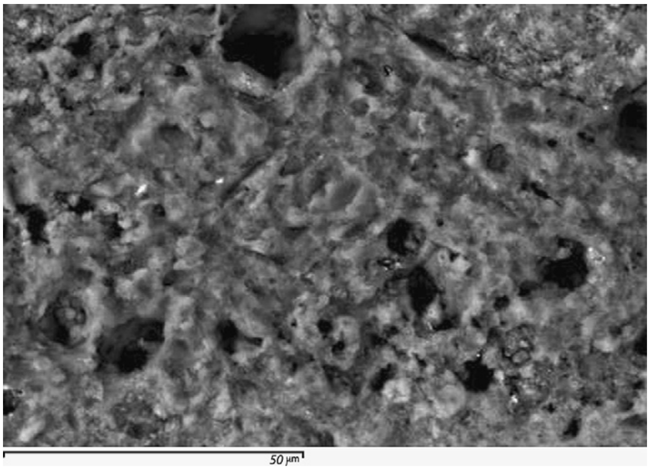

Fig. 6. Sample Ca31. Detail of isotropic layer in Fig. 5 (solidified volcanic ash) showing multiple vesicles. Scale bar: 50 μm.

both materials have the same optical properties, showing a homogeneous brownish-yellow colour under the plane polarized light and no optical activity under the crossed polars, as well as vesicles, which suggest that both materials correspond to fragments of solidified small-size particles of volcanic ash.

In addition to the presence of glass and acicular crystals, both of which suggest the presence of hydraulic compounds, hydraulic reactions were clearly observable as rims around isotropic materials in Sample Ca11. Analysis of the reaction rims showed that they have a composition of 51.7% SiO_2 and 46.2% CaO (see Table 2). This indicates the presence of a calcium silicate hydrate obtained through the use of a pozzolanic aggregate rich in reactive silica.

From a technological point of view, the possibility that pozzolanic plasters were utilized in Preclassic and Late Classic times at Calakmul suggests craft specialization and a good empirical knowledge of materials. In the Late Preclassic period, it is well known that Calakmul was an important site with a growing population and impressive architectural arrangements (Carrasco Vargas, 2000). In the case of the Late Classic period archaeological and epigraphic evidence demonstrate that Calakmul was a powerful site and a key player in political life (Folan et al., 1995; Martin and Grube, 2000). All of this suggests that during these periods there existed specialized and organized production that would explain the introduction and use of volcanic ash in the plasters. We recognize, however, that the sampling was restricted and further studies may reveal a more fine-grained picture of architectural practices at Calakmul.

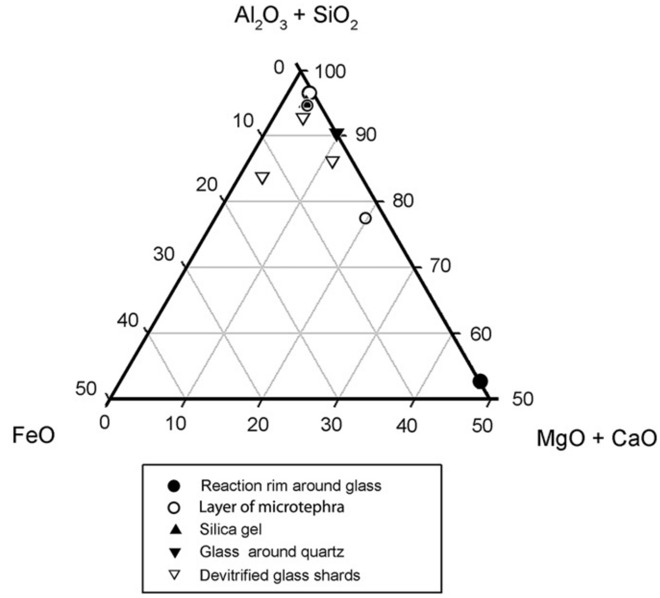

Fig. 8. Semi-quantitative elemental composition (major components) of isotropic materials and hydraulic reactions as measured by EDS analysis.

6. Conclusions

Based on the presence of acicular crystals, isotropic materials and silicon dioxide-rich particles, on occasion with the characteristic sickle shape of volcanic glass, we hypothesize that volcanic materials were deliberately added to the plasters at Calakmul and Lamanai in order to produce hydraulic compounds by mixing the lime with locally or regionally available volcanic ash. The deliberate exploitation of volcanic materials is also supported by the numerous reports that have been previously documented on the use of ash-tempered ceramics in the Maya lowlands, all of which suggests that there was a pattern in the exploitation and use of volcanic materials in the Maya lowlands.

In the case of Lamanai, the chemical alteration of the glass shards observed in Late Postclassic and Historic plasters seem to indicate the exploitation of geologically old deposits of volcanic ash, possibly pointing to the Bladen Volcanic Member. In the case of Calakmul, the characteristics of the isotropic inclusions observed in Preclassic and Late Classic samples suggest the use of solidified submicroscopic fragments volcanic ash. Based on the report of an ash layer by Gunn et al. (2002) at Calakmul, as well as the ash layer reported in this paper, it is possible that there were numerous ash

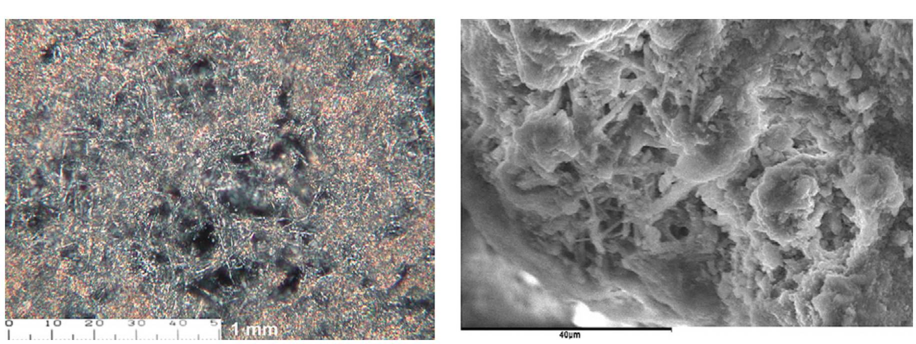

Fig. 7. Acicular (needle-shape) crystals. Left: sample Ca6, Middle Preclassic period, Calakmul. Transmitted cross polarized light, scale bar: 1 mm (10 units represent 100 μm). Right: sample Ca8. Late Middle Preclassic period, Calakmul. Backscattered electron image, scale bar: 40 μm.

falls and that this material was procured locally in the southern and central lowlands.

Although the use of volcanic materials has been proposed in the case of Maya ceramics, the use of reactive volcanic silica in plaster mixtures has an additional technological relevance, because it suggests that the ancient Maya were aware of the characteristics of volcanic ash and the benefits of pozzolanic plasters.

Further research is needed at Lamanai, Calakmul and other Maya sites to test the hypothesis that volcanic ash was employed in architectural plasters. Confirmation is problematic because the silica from volcanic ash reacts with the slaked lime, with the result that the pre-reactive components are difficult to identify. For this reason, further work needs to be done in order to obtain a more detailed identification of the hydraulic components by means of alternative analytical techniques, including the use of thermal and thermogravimetric analysis. Such techniques can identify specific compounds that are diagnostic of hydraulic and pozzolanic limes and that cannot be characterized by other techniques, such as X-ray diffraction, owing to their poor crystallinity. Chemical analysis, such as the pozzolanicity test and the quantification of soluble silica, also constitute relevant techniques for the characterization of hydraulicity (see Van Balen et al., 1999) and should therefore be employed in future research.

At another level, more plasters and related materials from the Maya area need to be analyzed. For example, the characterization of inner linings of cisterns or *chultunoob*' is of particular interest regarding the possible use of pozzolanic plasters for producing more durable renders and therefore improving the storage of water, as the technique was used in the Old World in antiquity.

Finally, more detailed geochemical analyses are needed in order to connect the volcanic materials observed in the plasters with specific volcanic eruptions.

Although more research is needed on the topic, this preliminary report aims at encouraging other researchers to sample and analyze more plaster and to consider that the use of volcanic materials in the Maya Lowlands may have been more widespread than previously thought.

Acknowledgments

We are indebted to Kevin Reeves and Philip Connolly, technicians of the Wolfson Archaeological Science Laboratories, UCL, for their assistance in the analysis of the samples. We also thank Ruth Siddall and Clifford Price, supervisors of the first author, for their professional input during this research. We are grateful to the archaeologists Ramón Carrasco Vargas, Omar Rodríguez Campero and Verónica Vázquez López at the site of Calakmul, and we also thank Scott Simmons, Laura Howard and David Pendergast at Lamanai for their help with the sampling. In addition we want to express our gratitude to the Instituto Nacional de Antropología e Historia (INAH) and the Consejo de Arqueología in Mexico, and to the Institute of Archaeology, National Institute of Culture and History, of Belize, for providing sampling and export permits. Finally, we would like to thank David Pendergast, Thilo Rehren, Linda Howie and the anonymous reviewers for their sensible comments and edits to this paper.

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