Seventh Canadian Masonry Symposium
McMaster University
Hamilton, Ontario June 4-7, 1995

DURABILITY OF 16th CENTURY MORTAR: ARCTIC CANADA VERSUS INLAND IRELAND

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ABSTRACT

The durability of mortar is dependent on its physical properties, as well as the environment to which it is exposed. Mortars taken from stone structures in Arctic Canada and inland Ireland, dating from the mid 1500's, are examined using petrographic and scanning electron microscopy, mercury porosimetry, and thermal gravimetric analysis. Their apparent durability is probably more dependent on their location, rather than their inherent properties.

INTRODUCTION

Lime-based mortar has been used in masonry structures for at least 3000 years. Vitruvius' Ten Books on Architecture , written in 25 B.C. is considered the first manual for lime mortar and rendering usage and little has changed in the fundamentals of mortar preparation and placement over the years. Troublesome, however, is the wide range of durability of mortar. Some have lasted 3000 years and others not a single season. Investigating old mortars which appear to show good performance, may provide therefore, some understanding of what factors affect durability.

This study will present data on two mortars dating from approximately the mid 1500's. The first is from a stone structure built to house the members of the Martin Frobisher 1578 expedition to Kodlunarn Island off Baffin Island in the Canadian Arctic. Samples

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were provided by Dr. Reginald Auger, CELAT, Faculté des Lettres, Université de Laval. The second is from the Drum Roe Castle, Kilkenny, Ireland, built in the mid 1500's Samples provided by Dr. P. E. Grattan-Bellew, National Research Council, Institute for Research in Construction.

APPROACH

The traditional approach to analysis of historic mortars has been to use a chemical technique (Jedrzejewska, 1960, Cliver, 1974), whereby, the mortar is divided into a soluble, sand and cement fraction. These data can give an estimate of amount of carbonate and degree of carbonation, amount of aggregate and possibly mix proportions,. The presence of impurities might indicate the use of cement or a pozzolan. Not included in this approach, is a detailed assessment of the aggregate mineralogy, or paste, the nature of deterioration of the mortar, or an assessment of the microstructure. Chiari *et al.* (1994) suggest that Thermal Gravimetric and Differential Thermal Analysis (TG-DTA) and X-ray Diffraction (XRD) techniques, supported by hand sample, thin section descriptions and mercury porosimetry provide a means of evaluating the nature of historic mortar. This paper reports the results of the second approach.

The TG-DTA analysis was run on a Thermal Analyst 2100 - TA Instruments - 951 Thermogravimetric Analyser. The heating rate was 20°C/ min. over a temperature range of 20° to 1000° C. The analysis was conducted in nitrogen gas. Only the thermal gravimetric data will be presented. It represents changes in mass that can be related to the evolution of gases such as water vapour, CO_2 and SO_2. Crushed samples of the whole mortar were analysed. The samples were not dried prior to analysis.

The XRD was completed on a Rigaku Giegerflex powder diffractometer. The X-ray source is a Cu-tube, and the 2θ range was from 2° to 50°. Powder mounts consisting of the whole mortar were analysed.

Polished thin-sections (30 µm thick) were prepared from epoxy-impregnated samples of mortar. The thin sections were examined using a transmitting light petrographic microscope and a Cambridge 250 scanning electron microscope (SEM) with a Robinson back scatter emission (BSE) detector.

Mercury porosimetry was completed on samples of mortar in a Autoscan 33, Quantachrome porosimeter. The number 33 refers to the maximum intrusion pressure of 33,000 psi. The samples were immersed in isopropanol for 7 days, to displace all the water, dried in a vacuum oven for 1 day and stored in a desiccator until analysed.

RESULTS

Frobisher Mortar

Hand Sample Description. The mortar was collected as 25 mm to 50 mm diameter samples. It has a buff-white hue, with no significant differences in colour on any surface. Distingushing exterior or exposed surfaces from interior surfaces is difficult. Two distinct surface types are noted. One is smooth with a deposit of calcite and the other is rough with a sandy feel due to exposed quartz aggregate. Sub-spherical voids, up to 2 mm in diameter and up to 1 mm deep, pock 30 % of both surfaces. The interior of the voids are smooth suggesting a secondary mineral deposit, representing the location where aggregate has fallen out. Black specs of lichen, 0.5 mm in diameter, dot 10% of the rough surface, indicating that these surfaces were previously exposed to the elements at one time.

The aggregate has two size populations represented by different mineralogy (Table 1) and grain size (Table 2). Cream white carbonate is the most abundant coarse aggregate. It ranges in diameter from 1 mm to 10 mm with 2 mm the mode. The carbonate is very fine grained and soft. Light brown coloured flint occurs as elongate (up to 10 mm long) flakes. Quartz-biotite-plagioclase gneiss is the coarsest aggregate, ranging from 10 to 50 mm diameter. This aggregate is highly angular. Relatively rare, are pieces of charcoal, 2 to 5 mm diameter, interspersed in the mortar.

Red surface stained quartz is the most abundant fine aggregate. It is sub- to well-rounded and ranges in diameter from 0.5 to 1.0 mm. Black flakes of biotite, grains of green hornblende and a well rounded green mineral, interpreted to be augite, are similar in diameter to the quartz, but less abundant.

Table 1. Modal abundance of aggregate.

Frobisher Mortar			Drum Roe Mortar		
Aggregate	Modal Abundance (%)	Grain Size (mm)	Aggregate	Modal Abundance (%)	Grain Size (mm)
carbonate (chalk)	35	1 - 10	quartz	70	0.5 - 1.0
quartz	35	0.5 - 1.0	muscovite	15	0.5 - 2.0
flint	10	1 - 10	composite	10	0.5 - 8.0
biotite	5	0.5 - 1.0	brick	2.5	0.5 - 0.8
biotite gneiss	10	10 - 50	carbonate	2.5	0.5 - 1.5
hornblende	2	0.5 - 0.8			
augite	1	0.5 - 0.8			
charcoal	2	2 - 5			

Table 2. Sieve size analysis of Frobisher and Drum Roe mortar.

Sieve Size (μm)	Percentage Retained	
	Frobisher	Drum Roe
1000	16.76	46.90
600	3.35	12.69
300	24.58	12.29
212	25.95	5.83
150	20.11	3.44
<150	9.22	18.84

Thin Section Description. The thin section provided confirmation of the identification of the minerals noted in the hand sample, and identified the black and green minerals as hornblende and augite. In addition, information was obtained on the relative relationship of the aggregate to lime paste, and the textures within the paste. The aggregate makes up 40% of the mortar. It is well dispersed in the paste, with few grains touching. Aggregate paste, boundaries, for the most part are interrupted, with cracks. The carbonate grains are fossiliferous, identifying them as a limestone. Distinguishing the boundary between the carbonate aggregate and paste is difficult, and is often identified based on subtle changes in texture.

The paste is composed of calcite and makes up 60% of the mortar. It shows two broad textural forms: dense(25%) and porous (35%). Each occur in irregular patches, and are transitional from one to another (Fig. 1a). Elongate, ameboid-shaped voids, up to 500 μm long and 50 μm wide occur within the dense paste. The voids are not substantially interconnected. Fine crystals of calcite may line the voids, which represent the smooth surface noted in hand sample. The exterior surface occurs as a thin (20 μm) wide deposit of calcite cystals where it is a dense paste. This accounts for the smooth texture (Fig. 1b).

The porous texture appears marginal to aggregate and is dominantly pore space and calcite. There is some suggestion in Fig. 1a of a second phase. It has not been identified.

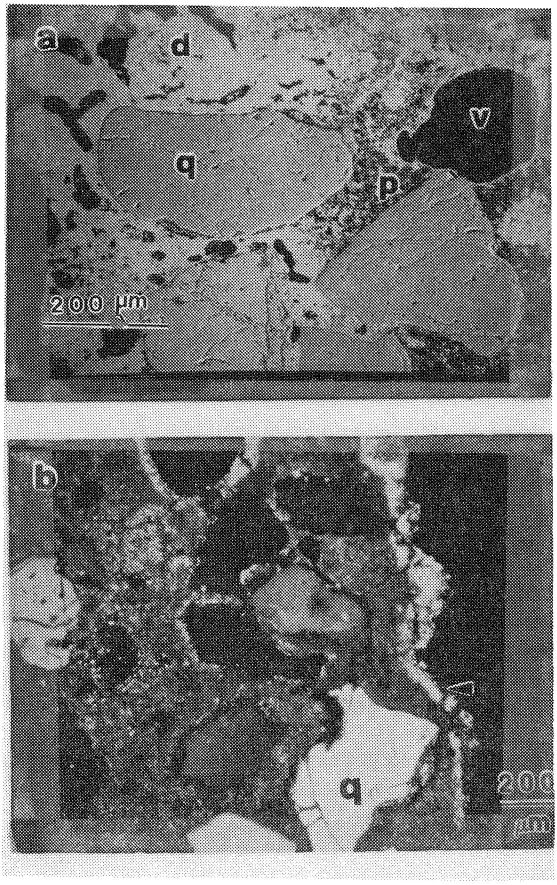

Figure 1. Frobisher Mortar. **a)** SEM, BSE photomicrograph illustrating round nature of quartz aggregate (q), dense paste (d) and porous paste (p). Voids (v) are irregular in shape. **b)** Transmitted light microscope photomicrograph (crossed polars) showing exposed edge of mortar. Arrow points to relatively thin deposit of calcite on surface. Quartz aggregate (q) surrounded by paste.

X-ray Diffraction. Quartz and calcite peaks dominate the diffractogram (Fig. 2). The identified minor peaks are consistent with the presence of feldspar. Minor minerals such as biotite are not represented due to their relative low abundance.

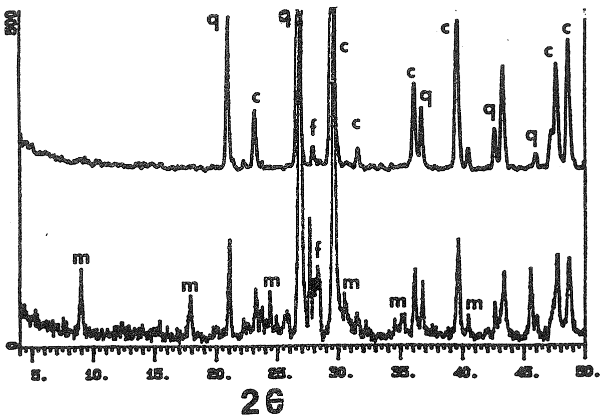

Figure 2. X-ray diffractogram of Frobisher (upper) and Drum Roe (lower) whole mortars. Dominant peaks are quartz (q), calcite (c), with muscovite (m) and feldspar (f) as minor peaks.

Thermal Gravimetric Data. The TGA pattern shows a large change in weight at about 750°C which is consistent with the evolution of CO_2 from calcite. The calculated value of calcite is 57%.

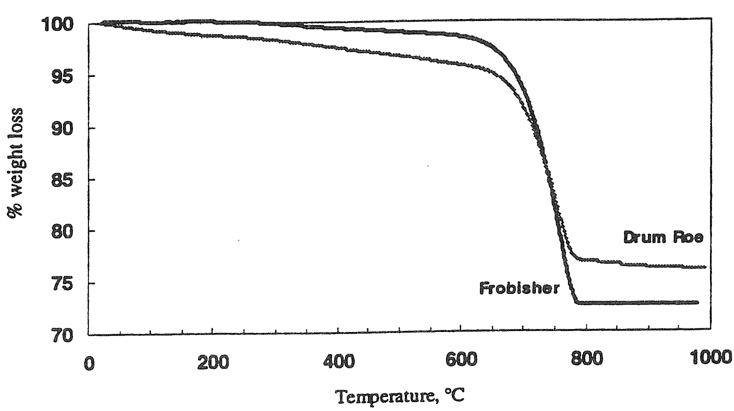

Fig. 3. Percent weight loss.

Mercury Porosimetry. The cumulative pore size distribution of the Frobisher mortar is presented in Figure 4. The total effective porosity, those pores which could be filled, represent 23% of the mortar. The bulk of the pores, 18%, occur in the range from 10 μm to 0.08 μm.

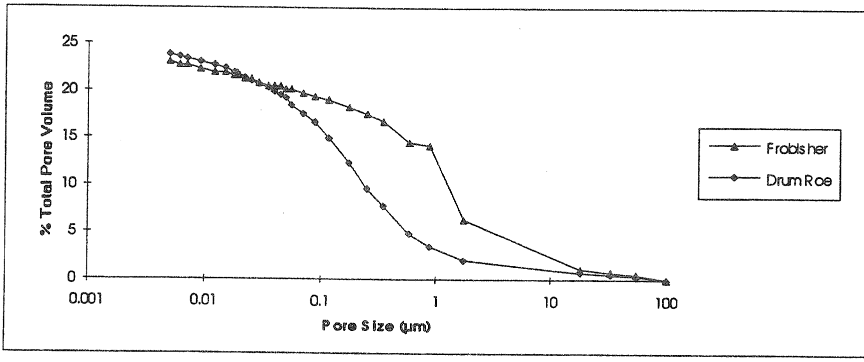

Fig. 4. Cumulative porosity of mortars.

Drum Roe Mortar

Hand Sample Description. The mortar was chiseled out of a stone wall, and taken in fist sized (90 mm wide) samples. The exposed surface of the mortar has a distinct speckled, mid-grey hue. Green moss grew over 10% of the exposed surface. The surface is highly irregular, characterized by impressions up to 40 mm across and 10 mm deep, which may be lined with a creamy white mineral deposit, suggesting locations where aggregate has fallen out. The interior surface has a buff tone, and is soft. A fine dust is lifted when rubbed.

The aggregate is dominated by a coarse grained population (Tables 1 and 2). Creamy white carbonate is the most abundant, and coarsest, ranging in size from 9 to 10 mm in diameter. White mica, 1 to 2 mm in diameter, is evenly distributed in the paste. Quartz and rock aggregate are more difficult to see in the interior of the paste and best seen on the exposed, weathered surface. The size ranges from 0.5 mm to 3 mm. Throughout the mortar an ocher coloured aggregate is visible. It is clay brick or tile.

Thin Section Description. The thin section examination confirmed the identification of the mineralogy of the aggregate from the hand sample. Aggregate makes up 50% of the volume of the mortar. The composite grains appear to have been derived from crushed rock, granitic source, but are highly altered by clay minerals and internally fractured suggesting weathering prior to use as a aggregate. The carbonate aggregate is fossiliferous, confirming its source as a limestone.

The paste comprises 50% of the mortar and is composed of calcite. It shows two broad textural forms: dense (30%) and porous (20%). The dense paste occurs as 50 μm wide rims to elongate, irregular shaped voids (Fig. 5a). The voids are interconnected and extend up to 5 mm in length. Well shaped calcite crystals rim the interior of the voids. The surface exposed to the elements is encrusted with a thick (50 to 100 μm wide intergrowth of organic material and calcite (Fig. 5b).

The porous paste is marginal to the aggregate, and the texture is poorly defined (Fig. 5a).

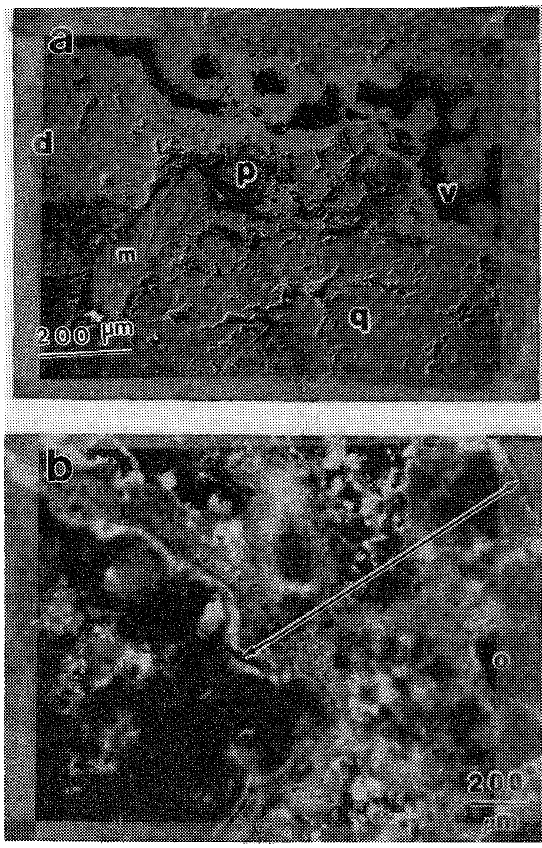

Figure 5. Drum Roe Mortar. **a)** SEM, BSE photomicrograph showing dense (d) calcite lining void space and porous (p) paste against quartz aggregate (q). Voids (v) are elongate and interconnected. Large cluster of muscovite blades (m) well illustrated. **b)** Transmitting light microscope photomicrograph (crossed polars) showing exposed edge of mortar. Arrow shows edge of surface carbonate and organic (o) intergrowth .

XRD. The diffractogram for the crushed whole mortar is presented in Figure 2. Quartz and calcite dominate the spectrum, but muscovite and feldspar are also present.

Thermal Gravimetric Data. The TGA pattern is presented in Figure 2. The loss in weight at 756°C is attributed to evolution of CO_2 from calcite. The calculated calcite content is 41%.

Mercury Porosimetry. The cumulative pore size distribution is presented in Figure 3. The total porosity is 24 % with the bulk of the pores, 18% between 2 μm and 0.08 μm.

DISCUSSION

Composition.
The compositon of both the aggregate and paste of the two mortars is fairly distinct (Table 3). This reflects the source of the aggregate and perhaps different techniques in preparing the mortar.

Table 3. Summary of analytical data.

	Frobisher	Drum Roe
dominant aggregate	40% - local beach sand, crushed rock and flint mixed with calcined limestone or chalk	50% - local weather granite
lime source	- calcined chalk limestone transported from England	- calcined local limestone
deterioration	- limited surface erosion due to lichen, aggregate loss, minor calcite deposition	- significant surface erosion due to lichen and moss, aggregate loss, significant calcite deposition
pozzolan	- none evident	- crushed brick

The source of the Frobisher mortar quartz and biotite gneiss aggregate is from locally derived beach sand and crushed rocks. The quartz is well rounded, consistent with a beach source. The use of biotite gneiss is consistent with the description of the local geology. The flint is not local, however, and was probably transported from England with the lime. The Frobisher expedition sailed from London, suggesting that the chalk and flint rich cliffs south-west of London, were the source.

The source of the Drum Roe mortar aggregate is local. The local geology consists of granite intrusions into limestone. Local history indicates that sand for mortar was removed from the weathered tops of granite intrusions. Lime was also locally calcined, in small kilns. The addition of crushed brick suggests that a hydraulic effect would have been produced. It is consistent with techniques dating from the Roman time, thought by some to have been abandoned in Europe during this time period (Ashurst, 1983).

Durability

The higher proportion of paste in the Frobisher mortar, relative to that of the Drum Roe mortar, suggests that the mortar may have been a richer mix and perhaps more workable. The rounded, and finer aggregate of the Frobisher mortar would also have made it more workable. The high clay content of the Drum Roe mortar, which would have had a relatively high water demand would have reduced its workability. The high lime content and high clay content, would suggest that neither mortar would have survived many cylces of saturatation and freeze-thaw cycling early in their placement history. The lime would have taken some time to carbonate, and therefore, gain enough strength to withstand the mechanical demands of freeze-thaw cycling. The high clay content may indicate that the aggregate-paste boundaries could have been contaminated by the clay size particles, again limiting the potential strength gain.

Each mortar shows some degree of mechanical and chemical deterioration. The Frobisher mortar shows cracks near the surface, which cross-cut the calcite deposits indicating they are relatively young, postdating carbonation. Aggregate has popped-out suggesting that the paste to aggregate bond became too weak to hold the exposed aggregate. The surface nature of the mechanical deteroration, might suggest that the cause is local saturation with water, and limited freeze-thaw damage.

The elongate voids in both mortars have dense and crystalline calcite developed along the margins. This indicates that they provided a pathway for moisture transport in and out of the mortar. The Frobisher mortar, although thoroughly carbonated, does not show the same extent of deposition of calcite crystals on voids or the same surface weathering as the Drum Roe mortar. This is consistent with the climate to which either is exposed. The Arctic climate, is essentially that of a desert, and with few freeze-thaw cycles and few conditions where the mortar would have been saturated with water. The Drum Roe mortar, on the other hand is in a damp, temperate climate, again without a significant number freeze thaw cycles to cause significant damage. Neither mortar appears to have been exposed to severe, and repeated freeze-thaw cycling, although they were clearly exposed to sufficient water or humidity to carbonate the paste.

The pore structure of a porous material is critical in providing freeze-thaw cycling durability (Litvan and Serda, 1978). There is some aggreement in the literature that well distributed, smaller pores in the range of 0.5 μm provide the needed pore structure to withstand freeze-thaw cycling. The Drum Roe mortar has a greater population of smaller diameter pores than the Frobisher mortar, perhaps suggesting that it would withstand freeze-thaw cycling more readily than the Frobisher mortar.

Work done by Litvan and Sereda (1978), followed by the Smeaton Project (Teutonico 1994), and at IRC (work in progress), suggests that the ancient tradition of the adding crushed brick provides a mechanical means of creating a suitable pore structure. The Drum Roe mortar contains crushed brick and has the largest abundance of small diameter pores. Clearly, additional work needs to be completed to investigate the source of the smaller pores.

The greatest amount of mechancial damage in either mortar appears to have occured on the surface exposed to the elements. This appears to be due to the colonization by moss and lichen. It may consume calcium from the mortar, and produce organic acids which dissolve the calcite.

CONCLUSIONS

The Frobisher and Drum Roe mortars have survived 400 years exposure to the elements. It is suggested that it was not the physical nature of mortars which account for their suvival but rather the elements to which they were exposed. They were not severe in terms of water saturation and freeze-thaw cycling.

ACKNOWLEDGMENTS

Technical assistance was provided by Mr. Herman Schultz, Mr. Ed. Quinn and Mr. Gary Pollomark. Dr. P.E. Grattan-Bellew and Dr. J.J. Beaudoin are thanked for their comments.

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