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Medieval lime-gypsum mortars in Ireland.

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Abstract. Historic lime-gypsum mortars are rare in Ireland. Gypsum is hygroscopic and relatively soluble in water, hence common believe was that it would hydrate in the highly-humid Irish climate to quickly weather. The material sources and fabrication technology of original mortars in the Kells Round Tower, built c.9th-10th centuries are studied.

The results evidenced that hydraulic, gypsum-lime mortars were used to build the tower. Their binder is a mixture of lime and gypsum. Rather than mixing lime with gypsum, the binder was made by burning a carbonate-gypsum rock (probably a marl) at the high temperatures typical of traditional gypsum production (~800°C). The mortars are hydraulic, as indicated by the high-temperature transformations evidenced with petrography, whereby clay minerals and carbonate have turned into calcium silicates. The presence of calcium silicates (in aggregates and lime inclusions) indicate that the mortars are hydraulic, and that a carbonate-gypsum rock was burned over 800°C to produce the binder.

The mortars were made with local materials. The use of gypsum this early is surprising. However, there are gypsum mines near Kells, at a short distance that could have been easily sorted in historic times. The parent rock used to make the binder was probably a marl, quarried in the Carrickmacross-Kingscourt area, c.10 km North of Kells.

Despite the gypsum content, the mortars have endured the Irish climate for about 1,000 years because the composition of the parent rock and the high production temperature afforded hydraulic properties to the mortars, and hence an enhanced durability. No other instances of use of gypsum-lime mortars in medieval times in Ireland have been reported to date.

KEYWORDS : Lime-gypsum mortar, calcium silicates, hydraulic gypsum mortars, historic mortars.

1. Introduction

This paper studies the original masonry mortars and plasters from the Kells Round Tower, in County Meath, Ireland. Round towers are common in ancient Irish monastic sites. The round tower studied belongs to the Kells monastic settlement, founded by Saint Columba c. 550, on land that had been gifted to him by the King of Tara - Diarmait mac Cerbaill. Columba was exiled after the Battle of Cúl Dreimhne. However, the Abbey of Kells was re-founded in the early 9th century by monks from Iona (Scotland), and the round tower was erected in the 9th/10th century (Murphy et al. 2020).

Many historic and traditional lime mortars in Ireland have no hydraulic properties because, most Irish limestones (Carboniferous age) mainly consist of calcium carbonate with minor impurities, hence they would produce pure, calcium lime when calcined (Pavia and Bolton 2000). Similarly, historic lime-gypsum mortars are rare in Ireland, but common over the world. Gypsum is widely found on earth, it can be relatively easily quarried and processed, requiring simple technology and low burning temperatures. Hence, it has been used widely throughout history. Gypsum might be the first man-made cementing material ever produced (Sanz 2009). By the late Stone Age (Neolithic), about 12,000 years ago, gypsum was widespread and primitive production organised (Sanz 2009). According to Sanz, historic gypsum mortars were heavily used in Italy, Portugal, Spain and France during the Postmedieval period, in the Renaissance and the 19thcentury. The most important ensembles of monumental gypsum in the world are probably in Spain and Morocco. The Islamic, decorative gypsum plasterwork built by the Nasrid (Nazari) Kingdom in the city of Granada (c.13th century) is probably the most exceptional collection of architectural gypsum in the world (Rubio Domene 2022).

In Ireland, gypsum plasters were used internally in the Georgian and Victorian periods (from the 17thcentury onwards), but no instances of use of gypsum, in masonry mortars or plasters, in early historic times have been reported to date. Gypsum is hygroscopic (it absorbs ambient humidity), and it is relatively soluble in water (2000 mg/l *versus* 80 mg/l for calcite=carbonated lime -$CaCO_3$- at 20°C). The high hygroscopicity and water-solubility of gypsum would cause early damage to gypsum mortars in the highly humid Irish climate.

Figure 1. The round tower of the Kells Monastic site, Co. Meath. Photo by L. O’Reilly.

Traditional and historic gypsum production used parent rocks with significant calcium carbonate and clay mineral content which were fired at high temperatures - for example during 20 to 80 hours at 950°C - (Middendorf 2002, Arens 2002, Sanz 2009, Gárate 1999, Sanz Arauz and Sepulcre Aguilar 2022). At these high temperatures, the gypsum - $CaSO_4.2H_2O$- in the parent rock transforms into anhydrite - $CaSO_4$-; the clay minerals and silica can react forming calcium silicates; and the original calcium carbonate transforms into quick lime -CaO- (Sanz 2009). As a result of the high-burning temperature of traditional production, the original gypsum binder is anhydrite - $CaSO_4$- based. This anhydrous sulphate hydrates when water is added during mixing to form gypsum - $CaSO_4$ $2H_2O$ - which causes the setting and hardening of the paste. Furthermore, the calcium silicates (formed from the reaction of the clay with carbonate and silica), afford the binder a hydraulic set. In contrast, modern industrial gypsum production uses purer raw materials and lower firing temperatures (120-190°C) which transform gypsum into calcium sulphate hemihydrate - $CaSO_4$ 0.5 H_2O- or basanite, known as plaster of Paris which is used internally, and it is the basis for most industrial gypsum products currently in the market. The lack of hydraulic impurities and the lower firing temperatures result in materials of lower mechanical strength and a greater porosity which are more hygroscopic and water-soluble than the traditionally produced gypsum-lime materials.

In Ireland, the main gypsum deposits are in the Triassic mudrocks and marls located West of Carrimackross, County Monaghan, and around Kingscourt, County Cavan (Hegarty 2017). Historic records claim that these gypsum deposits begun production in the 1800s. However, these deposits could have been exploited at a much earlier time. In the early 1800s, a fruitless pursuit of coal in the Shirley estate (south Monaghan) led to the discovery and intermittent exploitation of gypsum which was mined as early as 1835 (McDermott 2009). The Mohaghan gypsum deposits are currently mined producing a variety of gypsum-based products.

2. Materials and methods

The round tower was built with local limestone masonry. Limestone is the bedrock in the area and there are several historic quarries nearby. A selection of mortars was sampled on site. Three internal plasters, three masonry mortars and one mortar from the wall core were studied with thin section petrography. On hand, sample, the mortars are weathered but maintain cohesion. The quality, condition and site arrangement of the mortars suggest that they are original. They show typical features of historic mortars including a secondary, encrusted surface developed due to weathering. The petrographic (or polarizing) microscope is a standard tool in material science. It is used to assess material composition and damage, and to identify sources of raw materials and construction technologies. Thin sections of the mortars were cut from representative hand samples. They were polished to the standard thickness of c.20 microns and examined with a petrographic microscope, using eye pieces of 2, 10, 20 and 40 magnifications, with both natural and polarised light. The specimens were examined with natural and polarized light using the light source below the stage. The mineralogical composition was analysed with X-Ray Diffraction (XRD) using the powder method, with a Phillips apparatus including a

PW1050/80 goniometer and a PW3313/20 Cu k-alpha anode tube at 40kV and 20mA. In powder diffraction, a randomly oriented, fine powder is required for phase identification. Therefore, fragments of the mortars were crushed into small pieces and sieved to remove the aggregate. The binder was then ground into a fine powder. All measurements were taken from 3 to 70 degrees (2θ) at a step size of 0.02 degrees/second. The mineral quantities determined are indicative as they were estimated using the relative intensities of the peaks in the XRD trace, and the detection limit is approximately 5%.

3. Results

The petrographic and XRD analyses indicate that the binder consists of gypsum –$CaSO_4 \cdot 2H_2O$- and carbonated lime- $CaCO_3$. The presence of the remains of partially calcined gypsum rocks are evident in the mortars. There are other common features as explained below.

Plasters. The aggregate includes gypsum, calcined gypsum rock, shale, siltstone and possibly greywacke (clay-rich sandstone) (figure 2). Some aggregates include abundant hydraulic calcium silicates (figure 3-4 and 6-7). The abundance of gypsum in the binder (figure 5); the presence of gypsum-rock fragments altered by high temperature and often transformed into calcium silicates (figure 3-4 and 6-7); and the presence of gypsum with different habits (microcrystalline and crystalline aggregates, and microcrystalline binder) indicate that a gypsum rock was used to make the plasters (figure 8-9). The occasional presence of anhydrite in gypsum grains indicates that some gypsum did not fully hydrate (figure 9). Lime particles occasionally show clay minerals, silica and calcium silicates (figure 6). This, and the calcium silicates inside rock fragments prove that the mortars have hydraulic properties. Microscopic analyses also evidenced that some gypsum is secondary, due to weathering. It appears on the surface, in fractures and voids, and likely originates from the weathering of the original gypsum used to make the plaster.

Masonry and wall core mortars. They share common features with the plastering mortars as they all contain gypsum and have hydraulic properties. The original carbonated lime is (occasionally) recrystallized due to weathering by dissolution. Common features include:

- The presence of abundant gypsum and carbonated lime in the binder (figure 10-11).
- Presence of hydraulic phases (reactive silicates) inside aggregates and in the binder, probably generated from the burning of a parent gypsum rock that originally included clay minerals, carbonates, iron oxides and silica (figure 12-13).
- Presence of lime particles with impurities of clay and silica (figure 14).
- Presence of aggregates of greywacke, and mudrock/marls including carbonates, clay minerals, and abundant iron oxides (figure 10 and 17). Some are altered by firing and contain reactive calcium silicates (figure 12-13).
- Presence of secondary gypsum. Occasional rombohedral carbonates the suggest the occurrence of dolomite in the parent rock. They appear more weathered than the plasters, including abundant secondary gypsum filling fractures and at the interface (figure 15-17).

With respect to the **sources of raw materials**, the nature of the aggregate and the binder suggests that the mortars were made with local materials. Several authors report the presence of greywackes, marls, mudstone/dolomite in the area. Visscher (1971) describes alternate strata of dolomitic clay marls and gypsum in nearby Keuper outcrops of gypsum and banded marls. Vaughan et al. (2004) describe the sequence at the Knocknacran gypsum quarry as follows: laminated, gently folded, red and buff, Permian gypsum forms the basement, this is overlain by dark green and red, highly altered, pyroxene–dolerite and a weathered, green greywacke.

Plasters

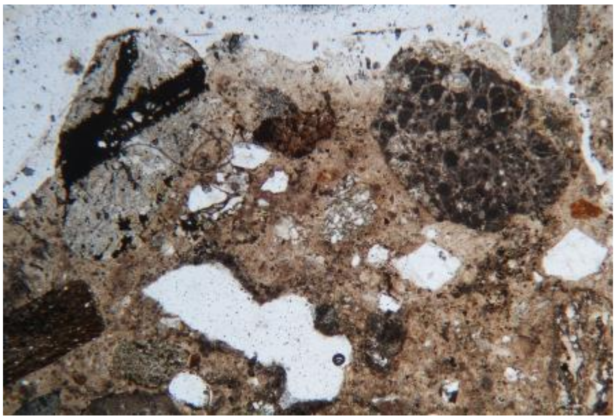

Fig. 2. General view of plaster A showing shale aggregate with pyrite and iron oxides (left), and a lime lump with hydraulic phases (right). 2X natural light. Field of view 7 mm.

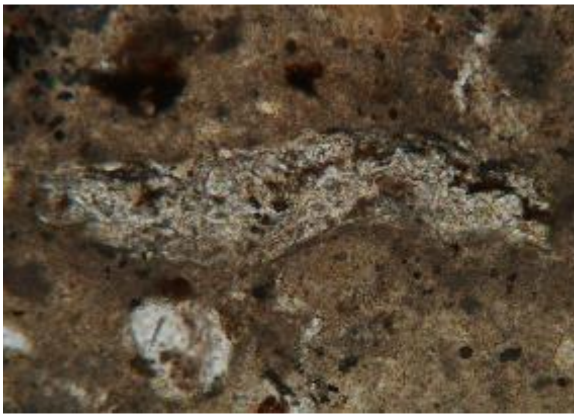

Fig. 3. Plaster A. Burned gypsum rock with hydraulic calcium silicates. 10X, natural light. Field of view 1.42 mm.

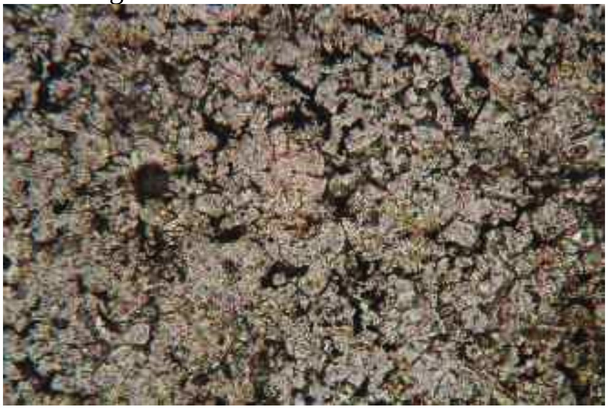

Fig.4. Detail of hydraulic calcium silicates in an iron interphase. 20X natural light. Field of view 0.7 mm.

Fig.5. Plaster A. Abundant microcrystalline gypsum (white) in the binder. 10X. Natural light.

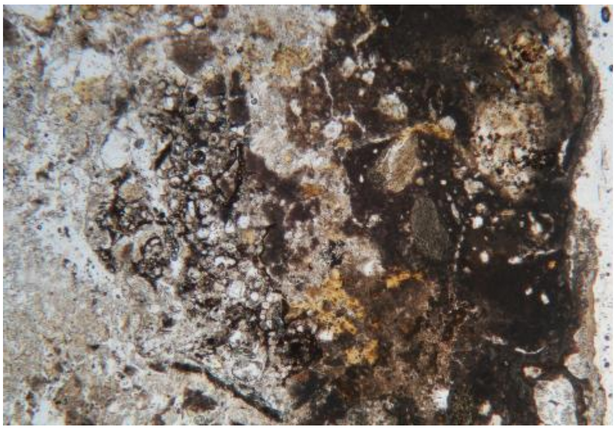

Fig. 6. Plaster C. Abundant hydraulic material in aggregate (left). 2X Natural light. Field = 7 mm.

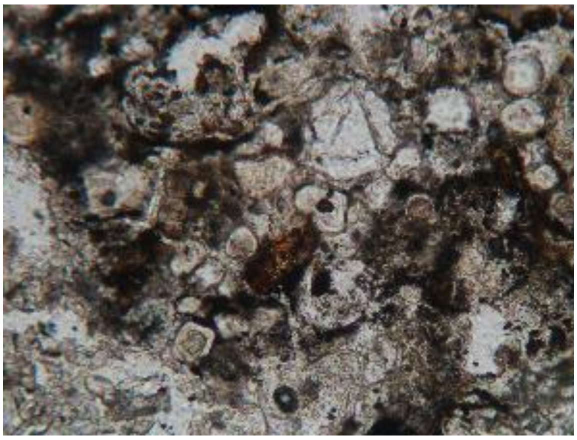

Fig. 7. Detail of the hydraulic material in figure 6. 10X Natural light. Field = 1.42 mm.

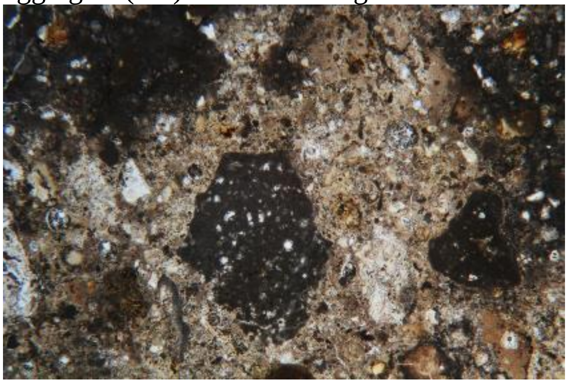

Fig. 8. Plaster B. Aggregates of gypsum (black and grey) and microcrystalline gypsum binder. Natural light 2X. Field of view 7 mm.

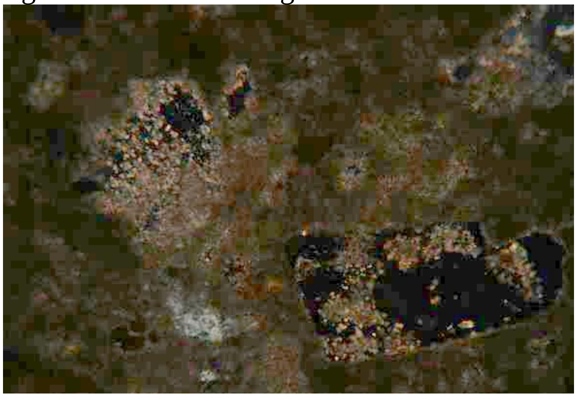

Fig. 9. Plaster B. Structure of two gypsum crystals including anhydrite. Polarised light. 10X. Field of view 1.42 mm.

Masonry and wall core mortars

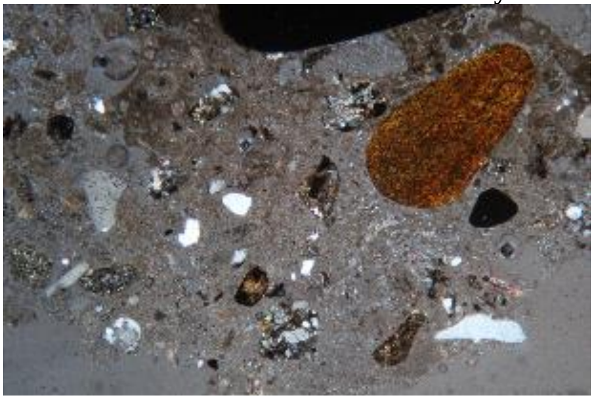

Fig. 10. Mortar 1: Abundant binder of lime and gypsum. Scarce aggregate including gypsum, quartz and rock fragments (with clay, oxides, carbonate). 2X, polarised light. 7 mm.

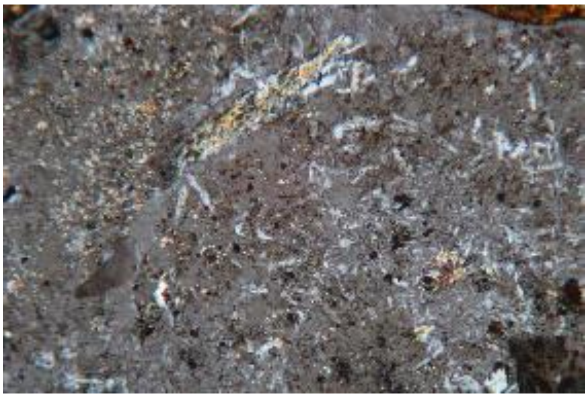

Fig.11. Binder of microcrystalline gypsum, occasionally lenticular or fibrous, and relicts of clay minerals (yellow). 10X, polarised light. Field of view 1.42 mm.

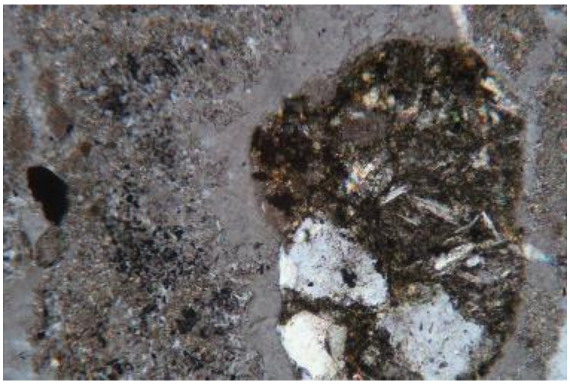

Fig.12. Aggregate of gypsum rock with clay and iron oxides. 10X, polarised light. 1.42 mm.

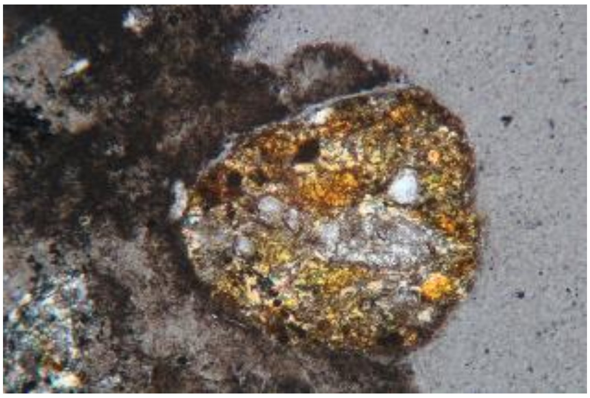

Fig.13. Aggregate including clay, iron oxides and newformed silicates. 10X, polarised light.

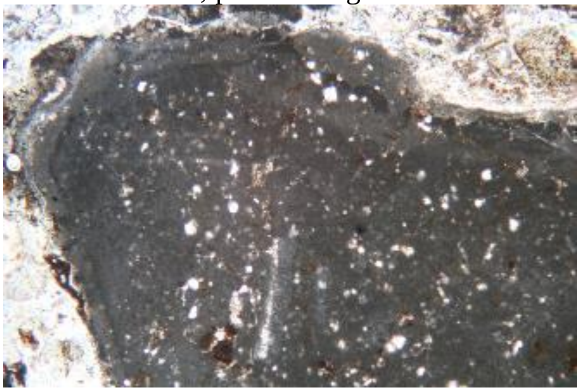

Fig.14. Coarse lime particle of carbonated lime with clay minerals and silica. 2X natural light. Field of view c. 7 mm.

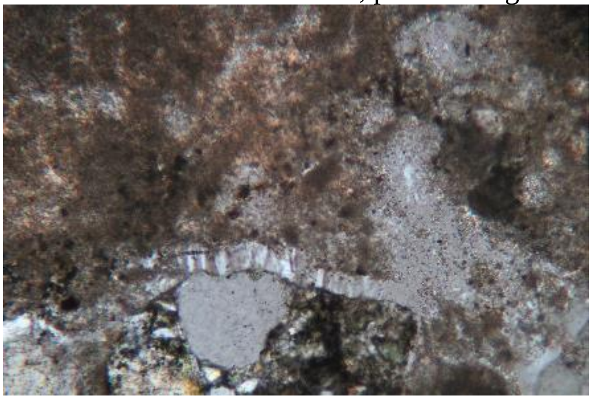

Fig.15. Secondary fibrous gypsum filling fractures and surrounding quartz at the interface. Gypsum scattered in the binder mixed with lime. 10X, polarised light.

Fig.16. Detail of secondary fibrous gypsum filling the interface around aggregate. 10X, polarised light. 1.42 mm.

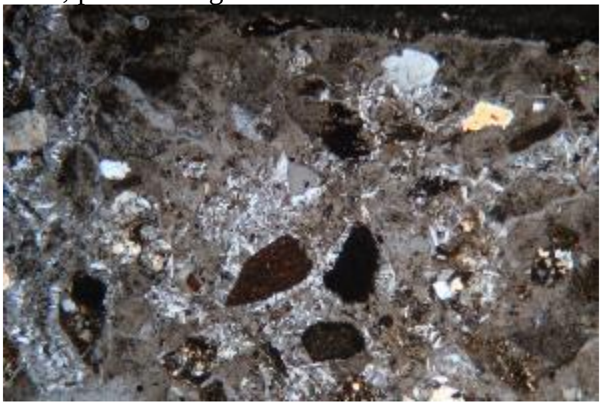

Fig.17. Gypsum binder and secondary fibrous gypsum in fractures. 2X left, polarised light. Field of view c. 7 mm.

The mineralogical analyses by XRD confirmed that gypsum – $CaSO_4 \cdot 2H_2O$- and abundant carbonated lime- $CaCO_3$ – form the binder of all the mortars studied. According to Sanz (2009), the most common calcium silicates in lime-gypsum mortars are larnite - Ca_2SiO_4 -, wollastonite - $CaSiO_3$- and Ca_3SiO_7. However, the XRD analyses did not allow to identify the precise nature of the calcium silicates seen under the microscope. Further research is set to determine the precise composition of these hydraulic silicates.

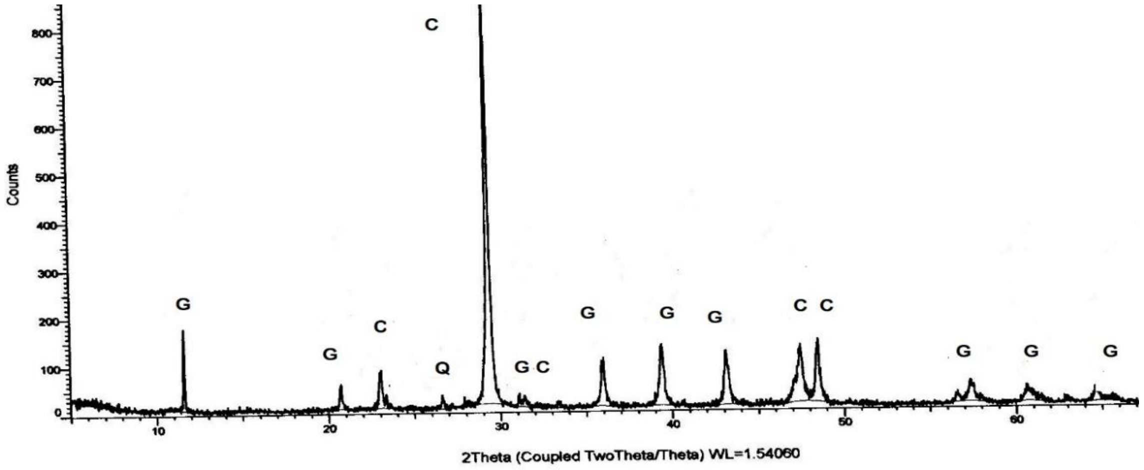

Fig. 18. Representative XRD trace including the mineral composition of the plaster's binder. C- calcite; G- gypsum.

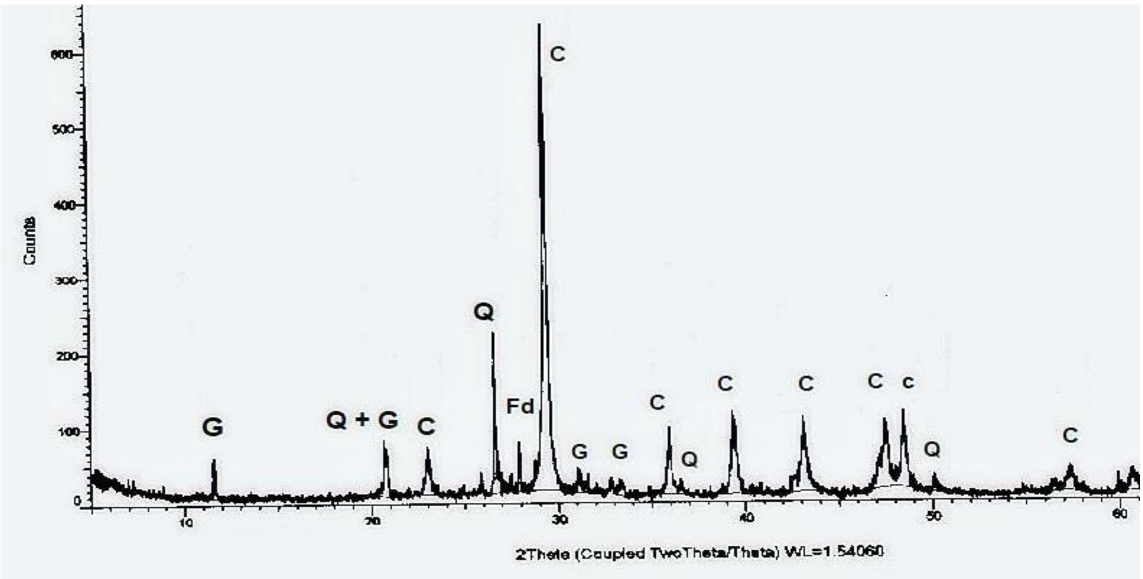

Fig. 19. XRD trace showing the mineral composition of the masonry mortar binder.
C- calcite; G- gypsum; Fd- feldspar.

4. Conclusion

Hydraulic gypsum mortars were used, in early medieval times (9-10th c.), to build the Kells Round Tower. The abundance and widespread distribution, texture and varying habits of the gypsum (granular, crystalline, microcrystalline, prismatic, lenticular) suggest that some gypsum is secondary due to weathering, but most is primary. The use of gypsum this early in Irish history is surprising and, to the author's knowledge, no other instances of use of gypsum in medieval times have yet been reported.

The mortars have demonstrated an outstanding durability, enduring the Irish climate for over 1000 years: their hydraulic binder is responsible for their good resistance to weather. The mortars have hydraulic properties, as evidenced from the presence of calcium silicates in partly burnt rock fragments, in lime inclusions and in the binder. Hydraulic properties are also inferred from the presence of clay minerals and silica in lime inclusions.

The binder consists of gypsum and carbonated lime. Two binders (lime and gypsum) could have been mixed to make the mortars, as reported in historic accounts in Spain, Italy and France. However, instead, the binder in the mortars studied was probably procured by calcining a carbonate-gypsum rock, a marl, at a firing temperature over 800°C. This is supported by the occurrence of partly-burned rocks including calcium silicates which have formed from the reaction of clay minerals with carbonate and silica at temperatures over 800°C, which agree with the typical burning temperatures of traditional gypsum production.

There are outcrops of marls interbedded with gypsum in the Carrickmacross - Kingscourt area, approximately 10 Km North of Kells. Historic records report that these deposits were mined from 1835 onwards. However, quarrying probable begun much earlier, and to sort this short distance would have been feasible in early historic times.

Acknowledgements

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References

Arens P. (2002) Untersuchung und Entwicklung von Gipsmörteln für den Außenbereich unter besonderer, Berücksichtigung der Wasserresistenz, PhD thesis Universität Siegen.

Gárate I. (1999) Artes de los yesos. Yeserías y Estucos. Madrid: Munilla-Lería.

Hegarty S. (2017) Monaghan's physical landscape: exploiting its natural resources. Geography Publications, 67-90.

McDermott M. (2009) Gypsum mining and the Shirley Estate. Four Courts Press, Dublin.

Middendorf B. (2002) Physico-mechanical and microstructural characteristics of historic and restoration mortars based on gypsum: current knowledge and perspective. Geological Society London, Special Publications, Vol. 205, 165-176.

Murphy C., O'Reilly L., Wall A. and Olohan I. (2020) Kells and District Tourism Network Headfort Rd, Kells, Co. Meath, A82 RY62

Raymond M. (1986) El enlucido exterior de yeso en las rehabilitaciones de las fachadas de París. *Informes de la construcción*, Vol. 38 (382), 35-39.

Rubio Domene R. (2022) In: Sanz Arauz D. and Sepulcre Aguilar A. (Eds) El yeso en la arquitecture historica. UPM Press. Univ. Politecnica de Madrid. ISBN: 978-84-18661-16-7

Pavia S. and Bolton J. (2000) Stone, Brick and Mortar. Historical Use, Decay and Conservation of Building Materials in Ireland Worldwell. Bray. ISBN: 9781869857325

Sanz Arauz D. (2009) Análisis del yeso empleado en revestimientos exteriores mediante técnicas geológicas. PhD thesis. Universidad Politécnica de Madrid. Escuela Técnica Superior de Arquitectura.

Sanz Arauz D. and Sepulcre Aguilar A. (Eds) (2022) El yeso en la arquitecture historica. UPM Press. Universidad Politecnica de Madrid. ISBN: 978-84-18661-16-7

Vaughan A. P. M., Dowling L. A., Mitchell F. J. G., Lauritzen S.-E., McCabe A. M. and Coxon P. (2004) Depositional and post-depositional history of warm stage deposits at Knocknacran, Co. Monaghan, Ireland. Journal Of Quaternary Science 19(6) 577–590

Visscher, H. (1971) The Permian and Triassic of the Kingscourt Outlier, Ireland. Geological Survey of Ireland Special Paper 1. 1–114.