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Magnesium oxychloride boards: understanding a novel building material

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Abstract Magnesium oxide type building boards are a relatively new alternative to traditional sheeting materials such as plywood, gypsum plasterboard and fibre-cement board. They have many advantages; strength, lightweight, ease of use and excellent fire resistance, which has become increasingly important as demanded by industry and required by more stringent legislation. Recently cases of durability issues associated with magnesium oxychloride boards in Denmark have emerged, however the precise nature of the problem was not established. These issues have been related to magnesium oxychloride boards which were exposed to high levels of moisture. In this paper the mechanism of the failures observed in Denmark has been investigated. The difference in quality between various magnesium oxychloride boards available in the market was also studied. It was found that there are significant differences, both physically

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L. Bagnall Resistant Building Products, 7 Duncrue Place, Belfast BT3 9BU, Northern Ireland, UK and chemically, between magnesium oxychloride boards supplied from different manufacturers. Crucially, the performance of each board when exposed to high levels of relative humidity was vastly different. Some of the boards investigated displayed behaviour similar to that observed in Denmark, whilst other boards exhibited substantial resistance to humid environments and had not deteriorated after 60 weeks of exposure.

Keywords Magnesium board · Magnesium oxychloride cement · Humidity · Water absorption · Permeability

1 Introduction

Magnesium oxychloride building boards, a relatively new sheeting material outside China, are used in the construction industry as an alternative to plywood, gypsum plasterboard, fibre-cement and other sheeting materials [1]. They are used in a variety of internal and external applications including sheathing, wall/ceiling linings, render carrier systems and prefabricated wall systems [1–3]. Magnesium oxychloride boards are typically produced from magnesium oxychloride cement combined with filler materials such as wood and perlite [4].

Magnesium oxychloride cement has been around for many years having been first reported by French



scientist Stanislas Sorel in 1866 [5]. It is based on a reaction between magnesium oxide (MgO) powder and magnesium chloride (MgCl₂) solutions [6]. Magnesium oxide powder is obtained from the extraction of magnesium carbonate (MgCO₃), sometimes referred to as magnesite. Almost 65% of the world's magnesite resources are found in China, North Korea and Russia [7]. Magnesite is calcined at temperatures greater than 600 °C to produce magnesium oxide [8]. The calcination temperature and duration has a significant influence on the crystal size, particle surface area and degree of reactivity of the resultant magnesium oxide [9-12]. Magnesium chloride is usually extracted from brines such as the Dead Sea and the Great Salt Lake followed by processing to obtain the necessary composition and concentration [13, 14].

There are two main hydrate phases (magnesium chloride hydroxide hydrates) responsible for the hardening and strength of magnesium oxychloride cement, these are $5 \text{ Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 8\text{H}_2\text{O}$ (5-phase) and $3 \text{ Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 8\text{H}_2\text{O}$ (3-phase) [15]. Other phases of magnesium chloride hydroxide hydrate known as 2-phase and 9-phase can also be formed during the reaction process, but are not stable at ambient temperature [16]. Additionally, magnesium hydroxide (Mg(OH)₂), sometimes referred to as brucite, is a phase which occasionally forms depending on the proportioning of raw materials [12].

Many advantages of magnesium oxychloride cements over other cements have been reported including rapid hardening, high strength, good fire resistance, low thermal conductivity and good resistance to abrasion [17-26]. Consequently, magnesium oxychloride boards have many advantages when compared with traditional board types such as plywood, gypsum plasterboard, fibre-cement and oriented strand board (OSB). For example, they are less dense than fibre-cement boards making handling easier for construction workers and reducing loads on buildings [27]. Additionally, cutting magnesium oxychloride boards does not require sophisticated or expensive equipment, a simple 'score and snap' method can be applied which requires only a knife. They are also much less susceptible to mould growth than boards made solely from wood. One of the key advantages of magnesium oxychloride boards is their fire resistance which is considerably better than gypsum plasterboard [28, 29]. Magnesium oxychloride cements have also



generated interest due to their lower environmental impact when compared with Portland cement. This is due to the adoption of a lower calcination temperature for light-burned magnesite (600–1300 °C) compared with the calcination of cement clinker (approx. 1400 °C) [19, 30, 31]. Additionally, Power et al. [32] has suggested that approximately 20–40% of the CO₂ emissions associated with the production of magnesium oxychloride boards may be offset due to passive carbonation over a 15 year period. Magnesium oxychloride boards are also considered less toxic to humans than conventional resin-based particle boards [33].

Whilst magnesium oxychloride boards offer many virtues, recent reports [34-36] have highlighted durability issues with magnesium oxychloride wallboards used in Denmark. Specifically, during periods of high relative humidity (RH) water droplets formed on the surface of the boards. These water drops have been described as 'tears' and the process has been termed 'crying' or 'sweating'. It has been claimed that the moisture caused mould growth and staining on building elements. Additionally, metal fixtures and fittings had corroded due to high soluble chloride content within the water droplets. In some instances the phenomenon also appeared to cause structural damage to the boards themselves as they were significantly cracked and damaged. It is not clear whether these failures are an inherent problem with magnesium oxychloride boards or were caused by an isolated quality issue with certain boards or due to poor construction details. This paper aims to assess the variability within magnesium oxychloride boards from different sources, to determine the process behind the observed issues, and to assess if a quality issue in the manufacturing process could account for the failures reported in Denmark. Recently a steering group led by The British Standards Institution (BSI) has been set up along with the Magnesium Oxide Building Board Trade Association (MOBBTA) to develop a Publicly Available Specification (PAS) document for magnesium-based construction boards.

2 Experimental methods

2.1 Board samples

Six magnesium oxychloride board samples were obtained for analysis and comparison. Each board sample was available in the UK market and was produced by a different manufacturer. These six boards were believed to cover more than 90% of the magnesium oxychloride boards sold in the UK in 2018. Each board was marketed as mainly consisting of magnesium oxychloride cement and also contained wood and perlite, as fillers, and layers of glass fibre mesh as reinforcement. As this is an investigative study exploring the properties and variability of industrial products, the exact proportions of raw materials used in each board is not known. Each board sample is identified as A-F in Table 1 and their nominal thickness and density is also shown. The density was obtained according to BS EN 323:1992 [37].

2.2 Testing procedures

2.2.1 Board composition

The composition of each board was examined by, X-ray fluorescence (XRF), X-ray diffraction (XRD), thermogravimetric analysis (TGA) with mass spectrometry (MS) and scanning electron microscopy (SEM). Powdered samples with a particle size ≤ 63 µm were prepared from each board for XRF, XRD and TGA. Samples were prepared for XRF using the fused bead method. XRD was carried out on powdered samples with a PANalytical X'Pert PRO diffractometer applying CuKa radiation of wavelength 1.54 Å.

 Table 1
 Thickness and density of six magnesium oxychloride board samples

D 1		D: (1 (3)		
Board	Nominal thickness (mm)	Density (kg/m ³)		
А	9.0	1092		
В	9.0	686		
С	9.0	1113		
D	9.0	993		
Е	11.0	1032		
F	12.5	998		

Diffraction patterns were obtained between 5 and 65° 2θ with a step size of $0.02^{\circ} 2\theta$. The mineralogy of the various samples was ascertained by using PANalyticals X'Pert Highscore Plus software in conjunction with the Powder Diffraction File database. Once the various phases were identified the crystallographic details of each phase was refined using the Rietveld method, within the Highscore software, to produce a pattern match. QXRD works on the principle that the area under the peaks is proportional to the amount of the phase producing it. TGA was carried out using Netzsch's TG 209. The temperature was increased up to 1000 °C at a rate of 10 °C/min in a nitrogen environment. Derivative thermogravimetric analysis (DTG) were used to confirm the presence of phases and hydrates. SEM was used to obtain images of the board's microstructure along their fractured surface. The equipment used was a JOEL JSM-6500F field emission scanning electron microscope.

2.2.2 Physical properties

The modulus of rupture, or bending strength, of each board was obtained using guidance from the method described in BS EN 12,467:2012 + A2:2018 [38]. Five rectangular specimens measuring 200×50 mm were cut from both the longitudinal and transverse directions and stored in ambient laboratory conditions for more than 7 days prior to testing. A three-point bending test was conducted with a span of 150 mm and a loading rate of 10 mm/min. The average of all 10 samples was then calculated to obtain the bending strength for each board.

The linear moisture movement of each board was obtained in general accordance with BS EN 12,467:2012 + A2:2018 [38]. One sample from each direction of each board was tested and the average calculated. The size of samples used was 250×75 mm. Each sample had two demec datum discs fixed to its surface 200 mm apart. The samples were conditioned at 23 °C and 30% RH until they reached constant mass. At this point the distance between the two discs was measured using a demountable mechanical strain gauge. The RH was then increased to 90% for 22 weeks and the distance between the two discs measured again. The linear moisture movement was then calculated as a percentage change between the two readings.

The water impermeability test was carried out in accordance with BS EN 12,467:2012 + A2:2018 [38]. A frame was fixed to the top face of each board and the area inside the frame filled with water to a depth of 20 mm. The underside of the board was then monitored for 24 h to assess whether any drops of water had formed below. The size of the board samples used were 600×500 mm and the size of the frame used was 550×450 mm. Only boards A, B C and D were tested as they each had an identical thickness of 9 mm.

The water absorption coefficient was obtained according to the partial immersion procedure given in BS EN ISO 15,148:2002 + A1:2016 [39]. Three samples measuring 100×100 mm were tested for each board. The outside edge of each sample was coated by epoxy paint to prevent any water ingression from the sides of the sample. The samples were then conditioned under test conditions (23 \pm 2 °C and $50 \pm 5\%$ RH) until two consecutive mass readings 24 h apart were stabilised to within 1% of the samples mass. The initial mass was then recorded and the samples were suspended in water with their top face down in contact with water. After intervals of 5 min, 20 min, 1 h, 2 h, 4 h, 8 h, 24 h and 48 h, the samples were removed from the water, the surfaces gently dried and the mass recorded. The samples were then returned to the water until the subsequent measurement. The water level in the container was also maintained. A graph of the change in mass over surface area is plotted against the square root of time and the water absorption coefficient calculated accordguidance ing to the in BS EN ISO 15,148:2002 + A1:2016 [39].

2.2.3 Accelerated ageing

In order to develop understanding of the mechanism of deterioration which occurs when magnesium oxychloride boards are exposed to moisture, three different accelerated exposure conditions were employed:

1. Samples of each board were ground into a fine powder with a particle size $\leq 63 \ \mu\text{m}$. Then 1 g of each powdered sample was added to a test tube with 25 g of deionised water. The test tube and contents were agitated for 24 h. Following agitation, the samples were centrifuged to separate the powder and water solution. The powders obtained



were then dried in a desiccator at 35 °C for 24 h before analysis by XRD. The water solutions were analysed using ion chromatography with a Dionex DX-310 IC system. Inductively coupled plasma mass spectroscopy (ICP-MS) was performed using an Elan 6000 system to obtain the content of metals. Certified reference standards were used to calibrate both instruments.

- Powdered samples of each board were placed into an environmental chamber and the conditions were maintained at 23 °C and 90% RH for 40 weeks. The powders were then dried in a desiccator at 35 °C for 24 h before analysis by XRD.
- 3. In order to evaluate how susceptible each board was to high levels of RH, a crying test was carried out on board samples (75 \times 75 mm). The edge of each sample was painted with epoxy paint so that only the top and bottom of each board sample was exposed to high RH. The samples were then placed in an environmental chamber for 60 weeks at 30 °C and 90% RH. The boards were monitored regularly to identify if and when crying had occurred. The samples were suspended inside plastic containers to keep water droplets from dropping directly onto them and being confused as crying liquid. These containers also allowed the crying liquid to be collected for analysis. The sides of the containers were perforated with 12 mm circular holes to ensure conditions inside the boxes and environmental chamber were identical. This was verified with a temperature and RH probe. After 60 weeks, samples of each board were powdered and dried in a desiccator with silica gel at 35 °C for 24 h before analysis by XRD. The crying liquid was analysed by ion chromatography and ICP-MS.

3 Results and discussion

3.1 Board composition

The chemical composition of each board obtained by XRF is shown in Table 2. The loss on ignition for each board is between 37 and 52%. This can likely be related to the wood fibre content of each board. XRF shows that the boards contain between 32 and 39%

Table 2Chemicalcomposition and loss onignition (LOI) of each board		SiO ₂	Al_2O_3	Fe ₂ O ₃	CaO	MgO	K ₂ O	TiO ₂	SO ₃	P_2O_5	Cl	Br	LOI
	A	13.61	2.41	0.22	1.79	35.15	0.42	0.03	0.77	0.12	8.00	0.07	37.40
obtained by XRF (wt %)	В	4.62	0.91	0.22	0.84	32.00	0.09	0.02	0.15	0.00	8.90	0.00	52.24
	С	9.63	1.52	0.20	1.27	35.98	0.25	0.02	0.78	0.00	8.19	0.08	42.06
	D	4.15	0.68	0.23	1.34	39.13	0.07	0.01	0.51	0.07	7.70	0.04	46.11
	Е	5.95	0.78	0.22	0.88	35.15	0.15	0.01	0.45	0.04	7.77	0.03	48.56
	F	5.95	1.10	0.14	2.41	33.33	0.16	0.01	0.60	0.04	8.53	0.05	47.66

MgO. As shown by XRD (Fig. 1) not all of this relates to MgO, rather to numerous other minerals which also contain magnesium. The main minerals identified by XRD are 5-phase magnesium chloride hydroxide hydrate $(5 \text{ Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 8\text{H}_2\text{O}),$ magnesite (MgCO₃), brucite (Mg(OH)₂), quartz (SiO₂), talc (Mg₃Si₄O₁₀(OH)₂) and calcite (CaCO₃). Board F also contains dolomite $(CaMg(CO_3)_2)$ which is in agreement with the increased calcium content present in board F (Table 2). In general, the same minerals are present in all six boards. However, there appears to be subtle differences in the quantity of each mineral. Table 3 shows the quantified values of each mineral. These values only account for crystalline materials, not the X-ray amorphous content such as the wood and perlite. The main binder component of each board is 5phase with 63.4% in board B and as much as 83.8% in



Fig. 1 XRD pattern of each board. 5 5-phase magnesium chloride hydroxide hydrate, B brucite, M magnesite, Q quartz, C calcite, P periclase, D dolomite, T talc

board C. The 5-phase mineral is the most desirable magnesium oxychloride phase as these crystals provide superior mechanical properties compared with other magnesium oxychloride cement phases [12]. Each board also contains magnesite which makes up between 4.3 and 16.8% of the binder of each board except for board B which has almost 30% magnesite in its binder. The presence of magnesite may be due inadequate calcination, in terms of temperature or duration, of the raw material (magnesite) [12, 40]. Brucite is present predominantly in boards A and D with as much as 27.6% in board D and 12.2% in board A. This is likely due to the use of greater amounts of magnesium oxide in the original mix design as suggested by the XRF data in Table 2. This indicates there was surplus MgO available than was needed to react with all of the MgCl₂ present meaning in theory no chloride ions would remain unreacted. The chemical formula below shows the chemical reaction which takes place to perfectly form 5-phase.

 $5 MgO + MgCl_2 + 3H_2O \rightarrow 5 Mg(OH)_2 \cdot MgCl_2$ · 8H₂O.

Li and Chau [12] discuss that excess magnesium oxide is often used in an attempt to ensure that no chloride ions remain free and unreacted. However, this approach should be taken with caution as unreacted magnesium oxide can cause dimension instability due to its hydration to brucite which occupies a significantly larger volume [8, 41]. The binder of board F consists of almost 10% dolomite which is much larger than the quantities (< 1%) found in each of the other boards. Other minerals such as talc, quartz and calcite are present in small quantities (< 2.5% of total crystalline content) in each board and could be due to impurities in the raw materials or the use of inert fillers. It is worth noting these values are only representative of the crystalline proportion of each board, as each board would have different quantities of



Table 3	Qua	ntitative	XRD
data for	each	board	

	5-phase	Periclase	Magnesite	Brucite	Talc	Calcite	Quartz	Dolomite
A	74.8	_	9.2	12.2	2.3	0.9	0.5	_
В	66.5	-	29.6	-	1.9	0.1	0.9	0.9
С	83.8	-	13.1	-	1.3	0.3	0.8	0.7
D	63.4	0.8	4.3	27.6	1.4	1.0	1.5	-
Е	78.2	-	16.8	-	2.4	0.2	2.4	-
F	74.1	-	8.5	5.7	1.5	0.3	0.4	9.5

amorphous fillers and the ratio between fillers and binder is expected to differ from one board to another. Boards B, C, E and F contain a significant quantity of possible inert Mg in the form of magnesite and dolomite, likely due to inadequate calcination of the raw material. This portion of Mg is not available to react with MgCl₂ and form magnesium chloride hydroxide hydrate phases. This may result in free unreacted chlorides in these boards if the manufacturers assumed that all of the Mg was in the form of MgO and available for reactions to take place.

TGA of each board was carried out and the derivative of the TGA traces (DTG) is shown in Fig. 2. The gas evolved during thermal analysis was analysed using MS. Generally, all 6 boards have



Fig. 2 DTG of each board sample

similar endothermic peaks but the locations vary slightly. Losses observed below 50 °C can generally be related to the release of non-chemically bound moisture. Between 100 and 200 °C the samples have either 2 (B, C and F) or 3 (A, D and E) endothermic peaks. Data obtained from mass spectrometry indicates that mass loss in this region was related to the release of H₂O molecules. This is in agreement with Luo et al. [42] who noted two main peaks in this region for magnesium oxychloride cements due to the release of crystal water from 5-phase in two steps forming 5 Mg(OH)₂·MgCl₂. They reported the first step between 50 and 130 °C and the second between 140 and 200 °C. Between 300 and 450 °C, 5 Mg(OH)2-MgCl₂ is thermally decomposed into MgO, also in two steps with the release of OH (300-350 °C) and HCl (350–450 °C) [43, 44]. The peak at around 450 °C can also be related to the decomposition of Mg(OH)₂ and the peak located between 500 and 550 °C is related to the release of CO_2 in the decomposition of MgCO₃ [42]. Board B has the largest peak in this region which is in agreement with findings from XRD which suggest board B has more magnesite than its counterparts. Finally, board sample F has an additional peak at approx. 600 °C which is likely due to the release of CO₂ from dolomite.

Figure 3 shows the morphology of boards A, B and C on fractured surfaces at magnifications of 1000, 5000 and 10,000. Due to the quantity of wood fibres, perlite and other possible fillers, interpretation is not as straightforward as it would be for pure magnesium oxychloride cements. Board A has pockets of well-defined needle-like structures which are more easily seen at higher magnification. These needles are 5-phase and are interlocking which contributes to the strength they provide [45, 46]. Tan et al. [47] reported that the addition of phosphoric acid resulted in a matrix with more needle like crystals on the fracture surface. It was not possible to identify needle like



Fig. 3 SEM images showing morphology of boards A, B and C

structures in boards B and C. This could be related to the phosphorus content of board A in relation to B and C (Table 2). Huang et al. [48] also agrees that the addition of phosphoric acid results in a well crystallised needle shaped 5-phase. Chen et al. [49] reported that 5-phase can also appear as plate-like crystals similar to those observed in board C. The morphology observed in board B appears similar to that of magnesite [17, 50].

3.2 Physical properties

The bending strength of each board is shown in Table 4. Board A had the largest bending strength of 23.0 MPa. This could be due to the well interlocked

 Table 4
 Bending strength and moisture movement of each board

Board	Bending strength (MPa)	Moisture movement (%)
A	23.0	0.07
В	5.3	0.25
С	17.8	0.13
D	9.2	0.05
Е	9.9	0.11
F	10.6	0.05

5-phase needle shaped crystals observed by SEM. Board C had a bending strength of 17.8 MPa. The



bending strength of boards D, E and F were all between 9.2 and 10.6 MPa. However, board B was much lower with a bending strength of only 5.3 MPa observed. In addition to the strength of the binder, the top and bottom surface of each board has layers of fibre glass mesh consisting of interwoven grids which appear to provide a significant contribution to the bending strength achieved. The grid size of the mesh used in board B appeared larger than for each of the other boards. The nature of the wood used may also be a key factor in the strength development of each board. According to Wang et al. [51] the type of wood and how it has previously been treated can impact on cement hydration reactions and adversely affect the strength development of cement particle boards.

Table 4 also shows the linear moisture movement for each board after 22 weeks. The samples were conditioned to constant mass at 30% RH prior to exposure to 90% RH. Therefore, it is anticipated that they would undergo some movement during early stages of exposure to 90% RH due to moisture uptake. After 22 weeks board B had a moisture movement of 0.25% which was much larger than any of the other boards. Boards C and E had a moisture movement of 0.13 and 0.11%, respectively. The moisture movement for boards A, D and F was between 0.05 and 0.07% after 22 weeks. This highlights the variability between different boards with boards A, D and F appearing less susceptible to moisture compared with the other boards, particularly board B.

The water impermeability test was carried out on boards A to D. Only boards A, B C and D were tested as they each had an identical thickness of 9 mm. According to the standard (BS EN 12,467), traces of moisture may appear on the underside of the board but there shall be no formation of water droplets within 24 h. This applies to all exposure categories except for boards which are intended for internal use. Table 5 describes the performance of boards A, B, C and D during the water impermeability test. Board C had the best performance with no traces of moisture on the underside after 24 h. Boards A and D both had traces of moisture which is acceptable according to the standard. Board B had traces of moisture after 2 h and large water droplets after 24 h (Fig. S1). This suggests board B has an increased porosity compared with each of the other boards and allows moisture to pass through more easily. It is worth noting that board B also has a much lower density (Table 1).

During 48 h of water absorption by partial immersion the mass change was recorded at various intervals. Figure 4 shows the mass change per area for each board over time as per the guidelines in BS EN ISO 15,148 [39]. By 48 h the mass of each board had generally stabilised indicating they had reached complete saturation. Board B absorbed water much faster than any of the other boards with much of its mass increase observed in the first 20 min. Boards D, E and F absorbed water more slowly but eventually absorbed a similar amount of water as board B. On the other hand, boards A and C absorbed much less water and at a lower rate.

According to the classification in BS EN ISO 15,148 [39], the graph for each sample will be type A or B and the water absorption coefficient (A_w) is calculated accordingly (Table S1). Boards A and C exhibited the best performance, with a water absorption coefficient of 0.0028 and 0.0026 kg/m² \cdot s^{0.5}, respectively. The water absorption coefficient for boards D, E and F was approximately 3 to 4 times larger $(0.0093-0.0132 \text{ kg/m}^2 \cdot \text{s}^{0.5})$. While the water absorption coefficient was much larger for board B $(0.0350 \text{ kg/m}^2 \cdot \text{s}^{0.5})$. It is worth noting boards E and F were thicker than each of the other boards (Table 1) meaning the sample volume was larger and therefore more space was available for water to be absorbed. These results indicate that board B is much more susceptible to water ingress than each of the other boards and is in agreement with the water impermeability test (Table 5). Findings also indicate that boards A and C absorbed the least amount of water and are the most resistant to the uptake of water. Boards A

Table 5 Performance of boards in water In water	Board	Comment on underside of boards	Pass/Fail
impermeability test	А	Traces of moisture after 24 h	Pass
	В	Traces of moisture after 2 h and large water droplets after 24 h	Fail
	С	Dry after 24 h	Pass
	D	Traces of moisture after 24 h	Pass



Fig. 4 Water absorption by partial immersion—change in mass over surface area against square root of time



and C also achieved the largest bending strength values (Table 4) and had the highest density (Table 1).

3.3 Accelerated ageing

The mechanism of crying has been investigated by accelerated board deterioration and observing board performance in environments with different levels of moisture available. Three different methods of accelerated ageing have been employed. The first method used was assessment of powdered samples, which were agitated in water for 24 h. The samples were finely ground, thus increasing surface area to volume ratio. Figure 5 shows the XRD patterns of the powdered samples following exposure to water for 24 h. There is a significant reduction in the quantity of 5-phase and there is a significant increase in the presence of brucite. The magnesite, quartz and calcite remain relatively unaffected. A similar trend was observed for each board. The only difference was how much of the 5-phase mineral had broken down after 24 h. For boards A and C the 5-phase mineral was completely broken down whereas for the other boards some 5-phase still remained. This is due to the testing procedure undertaken and not relevant to boards experiencing RH in-service. It is expected that continual breakdown occurred for boards A and C due to the lower pH of their water solution (Table 6), allowing the continuation of ion transfer between powdered board sample and water solution. In the case of boards B, D, E and F the pH of the water solution



Fig. 5 XRD patterns of each powdered sample following 24 h in water. 5 5-phase magnesium chloride hydroxide hydrate, B brucite, M magnesite, Q quartz, C calcite, D dolomite

was slightly higher which meant the water solution and powdered boards were in equilibrium and further ion transfers and breakdown of 5-phase were limited. It is stressed that this procedure is not relevant to real life exposure as wall boards are not submerged into a fixed volume of water in-service and the samples used were fine powder. However, this procedure was used to force the breakdown to occur in order to gain a better understanding of the process of chemical

 Table 6 Composition of leachate solution following accelerated board deterioration

Board	Concentration (mg/l)						
	Ca	К	Mg	Na	Si	Cl	
A	299	39.1	2738	15.3	0.37	6570	9.306
В	55.6	17.9	1582	3.57	0.27	3375	9.630
С	148	65.8	3086	19.2	0.42	6860	9.285
D	67.7	53.9	1368	21.8	0.14	2326	9.565
Е	109	52.9	1508	14.0	0.18	2675	9.665
F	36.3	73.2	1685	19.2	0.25	3025	9.642

change. In reality unmodified boards are exposed to intermittent periods of high RH rather than immersion in a fixed volume of water.

Table 6 shows the elemental composition and chloride concentration of the leachate solution following accelerated board deterioration. The main components that went into solution are magnesium and chloride. Small quantities of calcium, potassium, sodium and silicon were also found in solution.

The amount of magnesium and chloride is related to the amount of 5-phase broken down as shown by XRD. Boards A and C which had all of their 5-phase removed exhibited larger quantities of magnesium and chloride leached into solution. Based on the XRD patterns and the leachate obtained the mechanism of deterioration can be described as the decomposition of 5-phase by dissolution, allowing Mg²⁺ and Cl⁻ ions to go into solution with residual magnesium hydroxide being left behind:

 $5Mg(OH)_2 \cdot MgCl_2 \cdot 8H_2O$ $\rightarrow 5Mg(OH)2 + Mg^{2+} + 2Cl^- + 8H_2O$ [25]

This mechanism would account for the issues reported in Denmark [34, 35]. As well as the moisture from the environment the decomposition of 5-phase gives rise to the release of moisture previously contained within the 5-phase mineral structure. This moisture would be sufficient to facilitate mould growth and staining of building elements. The moisture released from 5-phase would also facilitate the transport of magnesium and chloride ions, allowing the chloride ions to corrode steel fixtures and fittings. Finally, the decomposition of a significant volume of



5-phase into brucite would result in structural damage to the boards themselves, a phenomenon observed in some instances in Denmark.

The second method of accelerated deterioration employed was the exposure of powdered samples to a temperature of 23 °C and RH of 90% for 40 weeks. Figure 6 shows the XRD pattern of each sample following exposure. For each sample, 5-phase and brucite are no longer present and hydromagnesite (4MgCO₃·Mg(OH)₂·4H₂O) had formed instead. The mechanism is slightly different than that for the powders exposed to water for 24 h. Hydromagnesite has formed due to the carbonation of 5-phase and the breakdown of MgCl₂. As shown in the formula below, the formation of hydromagnestite following the carbonation of 5-phase results in free water molecules and Mg²⁺ and Cl⁻ ions which could become dissolved in solution, forming a potential crying liquid containing Cl⁻ ions:

$$\begin{aligned} & 5 \text{Mg(OH)}_2 \cdot \text{MgCl}_2 \cdot 8 \text{H}_2 \text{O} \ + \ 4 \text{CO}_2 \\ & \rightarrow 4 \text{MgCO}_3 \cdot \text{Mg(OH)}_2 \\ & \quad \cdot 4 \text{H}_2 \text{O} \ + \ \text{Mg}^{2+} \ + \ 2 \text{Cl}^- \ + \ 8 \text{H}_2 \text{O} \end{aligned}$$

Power et al. [32] stated that hydromagnesite often forms through intermediary phases such as dypingite $(4MgCO_3 \cdot Mg(OH)_2 \cdot 5H_2O)$ and chlorartinite $(Mg(OH)_2 \cdot MgCl_2 \cdot 2MgCO_3 \cdot 6H_2O)$. Similar to the previous method of accelerated ageing, the 5-phase



Fig. 6 XRD patterns of each powdered sample following 40 weeks at 90% RH. *H* hydromagnesite, *M* magnesite



was broken down allowing the formation of other minerals which contain less water molecules. Therefore, this transformation of 5-phase to hydromagnesite could also easily account for the failures observed in Denmark. Whilst this method is useful to understand the mechanism of change when samples are exposed to high RH, it is not relevant to the performance of these particular boards in-service because the samples were powdered to accelerate their deterioration. Therefore, the third method of accelerated ageing will assess the performance of individual boards in a more realistic in-service environment.

To obtain a more realistic understanding of how the different boards may perform in-service a third method of accelerated ageing was employed. This involved a crying test on sections of each board exposed to a temperature of 30 °C and 90% RH. This test is more representative of in-service conditions than the previous tests carried out on powders and provides insight into the performance of different boards when exposed to high RH in-service. The crying time for each board is shown in Table 7. Boards B and C were observed to have cried by 11 days whilst boards F and E cried after 28 and 57 days, respectively. After 60 weeks boards A and D had not displayed any signs of crying. Figure 7 shows a photograph of each board after 60 weeks exposure, unexposed samples are also shown for comparison. Boards B, C, D and E have adopted a yellowish/brown colour whereas A and F remain almost white. There is a visible white deposit on the surface of boards C and F which was found to be the dypingite form of magnesium carbonate hydroxide hydrate. It is similar in structure to hydromagnesite, except it contains one additional water molecule.

The crying liquid for boards B, C and F was collected for chemical analysis. There was not enough crying liquid available to collect from sample E. The main components of the crying liquid were magnesium and chloride and the values are shown in Table 7. The ratio of the Mg to Cl found in the crying liquid (~ 0.34) is the same as the ratio of Mg and Cl found in MgCl₂. MgCl₂ is a key component of 5-phase $(5 \text{ Mg}(\text{OH})_2 \cdot \text{MgCl}_2 \cdot 8\text{H}_2\text{O})$. Therefore, this suggests the crying observed could be due to the breakdown of 5-phase. If the crying observed was due to the breakdown of 5-phase there should be a reduction in the quantity of 5-phase in boards B, C and F. The amount of Mg and Cl in the crying solutions was used to calculate the percentage of 5-phase breakdown the crying liquid for each board equated to. It was determined that the Mg and Cl in the crying liquids equated to 17, 3.5 and 7.8% of the 5-phase initially present in boards B, C and F, respectively. Figure 8 shows the XRD patterns of each board after 60 weeks exposure to 90% RH. In the case of board B, a reduction in the intensity of the 5-phase peaks was observed suggesting that the breakdown of 5-phase contributed to the crying observed. Boards C and F had minimal change to their XRD pattern meaning it is difficult to be certain the crying observed was due to 5-phase breakdown albeit the crying liquid only equates to < 8% of their initial 5-phase. Another possibility is that the crying was due to dissolution of amorphous MgCl₂ which is not observed by XRD. Gochez et al. [24] recently reported the presence of a small quantity of amorphous phases in magnesium oxychloride cements. Assessment of each boards initial composition also suggested that boards B, C, E and F may have residual unreacted MgCl₂ due to the amount of inert Mg in the form of magnesite and

Board	Crying time (days)	Mass increase (%)	Crying liquid volume (ml)	Magnesium (mg/l)	Chloride (mg/l)
А	-	26.8	-	-	_
В	11	59.9	5	20,000	57,783
С	11	17.8	3	16,500	51,132
D	-	27.8	-	_	-
Е	57	24.0	-	_	-
F	28	34.0	5	18,900	55,657

Table 7 Days until crying for each board during 60 weeks exposure to 30 °C and 90% RH and crying liquid composition





Fig. 7 Photograph of each board unexposed (left) and following 60 weeks exposure to 90% RH (right)



Fig. 8 XRD patterns of each board sample after exposure to 30 °C and 90% RH for 60 weeks. 5 5-phase magnesium chloride hydroxide hydrate, *B* brucite, *M* magnesite, *Q* quartz, *C* calcite, *D* dolomite, *T* talc

dolomite meaning insufficient MgO was available to react with all of the MgCl₂ present. It is interesting to note that these are the boards which also displayed crying. Boards A and D which did not cry had more



brucite initially, indicating that all of their MgCl₂ had reacted with MgO and surplus MgO was left over and formed brucite. This means it is unlikely boards A and D had any unreacted residual MgCl₂ available to go into solution and present as crying.

3.4 Discussion

The nature of the problems described in Denmark included the formation of water droplets containing chloride and structural damage to magnesium oxychloride boards. This study has compared the composition and performance of six different magnesium oxychloride boards. Three boards (B, C and F) have demonstrated behaviour similar to that observed in Denmark in terms of the formation of water droplets when exposed to high RH. The water droplets contained high levels of magnesium and chloride, consistent with the breakdown of MgCl₂. This is likely due to the breakdown of 5-phase, particularly in board B. The water droplets could also be due to incorrect proportioning of raw materials allowing unreacted soluble MgCl₂ to leach out when exposed to high RH. Therefore, it is likely that the failures observed in Denmark were due to either the breakdown of 5-phase magnesium chloride hydroxide hydrate or the presence of residual MgCl₂, or a combination of both. Alongside correct proportioning of raw materials, a homogenous supply of MgO is crucial. If the raw magnesite is not calcined consistently in terms of both temperature and duration, the resulting MgO will have differing levels of purity. This may allow the formation of additional excess brucite or detrimentally there may not be enough MgO available to react with the MgCl₂ allowing unreacted soluble MgCl₂ to remain in the boards. Another important part of the production process is the curing regime employed. Previous studies [16, 52] have found that the curing of magnesium oxychloride cement can have a significant impact on its moisture resistance. In particular board B displayed poor performance across all parameters investigated. It had the lowest bending strength, the largest moisture movement, lowest water impermeability and was most susceptible to water absorption. If such a board was used in Denmark it is no surprise durability issues were encountered. There is currently no standard documentation for magnesium oxychloride boards. It is important that such documentation is created in order to be able to set a minimum benchmark of durability performance required when formulating magnesium oxychloride boards.

Two boards (A and D) displayed superior performance in that no crying behaviour was observed after 60 weeks exposed to 90% RH. This appears to be due to correct proportioning of raw materials ensuring no MgCl₂ remains free and unreacted. Boards A and D may also have a more stable 5-phase magnesium chloride hydroxide hydrate when exposed to high RH. Recently numerous authors [24, 25, 47, 49, 53, 54] have reported that phosphorus provides a more stable magnesium oxychloride in the presence of moisture. When considering the phosphorus content of each board (Table 2), the boards with the largest phosphorus content are A and D suggesting the addition of phosphorus may have played a key role in their increased resistance to crying. The precise mechanism as to why phosphorus improves water resistance is not fully understood. One theory has been that phosphorus reduces the lowest concentration of Mg²⁺ ions required in solution, therefore stabilising the 5-phase [8, 25, 47]. More recently it has been suggested that as phosphorus is added the presence of a low water soluble amorphous phase increases. This amorphous phase partitions itself to the surface of 5-phase crystals protecting them from moisture [24]. Regardless of the exact mechanism, it has been proven that phosphorus increases the water resistance of magnesium oxychloride cements. Furthermore, this study provides a relatively good correlation between phosphorus content and time before crying occurs in magnesium oxychloride boards.

4 Conclusions

This paper highlights significant variability within magnesium oxychloride boards obtained from different manufacturers. The mechanism of the crying phenomenon observed in magnesium oxychloride boards in Denmark has also been investigated. The decomposition and dissolution of 5-phase magnesium chloride hydroxide hydrate was observed following different methods of accelerated ageing. The products left behind (brucite and hydromagnesite) following dissolution of 5-phase had less water molecules and chloride ions than 5-phase. Thus, a chloride rich solution is free to leave the boards and present as crying. It is worth noting that the accelerated aging procedures used in this study resulted in slightly different failure mechanisms. This suggests that no single accelerated test can reliably explain all the possible deterioration mechanisms for magnesium oxychloride boards. Alongside the breakdown of 5-phase, some boards appeared to have residual unreacted MgCl₂ which leached into solution and appeared as crying after exposure to high RH.

Two of the boards investigated (A and D) are significantly less susceptible to the crying phenomenon than their counterparts, which appear vulnerable to behaviour similar to that observed in Denmark. The likelihood of crying occurring for a particular board is likely related to a number of factors. Firstly, its chemical composition through correct proportioning of raw materials and the purity of those raw materials. Secondly, physical properties such as porosity which dictate how easily moisture is allowed to enter and pass through a board. Thirdly, the use of phosphorus which can help to protect 5-phase from dissolution. An adequate curing process is also likely to be key factor in order to allow the desired mineralogy to develop.

Significant differences between boards has been shown, in terms of composition, physical properties, and performance in humid environments. It is clear

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that not all magnesium oxychloride boards are the same and under real in-service conditions, the better performing boards have the potential to avoid failures, such as those observed in Denmark. There is an urgent need for recognised standards to distinguish products which are fit for purpose from those which are not. The development of the tests presented in this paper, alongside ongoing tests to determine service life and recognised standards, are crucial in order to avoid the recurrence of similar problems to those experienced in Denmark. Currently there is no standard documentation for magnesium oxychloride boards meaning it is difficult to establish a minimum performance level. The creation of specific standards would help differentiate boards that are fit for purpose from those which are not.

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Compliance with ethical standards

Conflicts of interest Grant obtained from Knowledge Transfer Partnership project and materials supplied by Resistant Building Products.

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