

Jan Erik Lindqvist, Timo Nijland,
Thorborg von Konow, Thale Sofie Wester Plesser,
Peter Nyman, Joe Larbi, Rob van Hees



Analysis of mortars with additives

SP Swedish National Testing and Research Institute

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Abstract

Existing methods for the analysis of masonry and rendering mortars were developed for the analysis of well defined simple mortars. Mortars used today are to a large extent hybrid mortars with different additives and filler. Analysing complex mortars with additives and fillers requires analytical routines that are more versatile. In order to investigate the possibilities for characterisation of mortars using chemical and microscopical methods, test prisms were produced from lime cement mortars with dolomite filler and from lime cement slag mortars. After six months of hardening, the prisms were prepared for chemical and microscopical thin-section analysis. Acid-soluble components in the samples were analysed chemically and the constituents were quantified by optical microscopy using point counting and counting in fields. From these results the mix proportions were calculated. The chemical methods gave an assessment of the mix proportions calculated using a general algorithm. The calculation of the quantitative results based on microscopy was done according to the NT BUILD 370 method and the TNO method. These gave a good assessment of the lime cement mortars and the slag mortars with low slag content. However, the analysis of mortars with high slag content gave aggregate-binder ratios that were too low. The obtained results show that the combination of microscopical and chemical methods can provide a good assessment of the proportions used when mixing the mortars.

Key words: mortar lime cement slag chemical microscopical

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**SP Swedish National Testing and
Research Institute**
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Postal address:
Box 857,
SE-501 15 BORÅS, Sweden
Telephone: +46 33 16 50 00
Telex: 36252 Testing S
Telefax: +46 33 13 55 02
E-mail: info@sp.se

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Preface

The mortars used for masonry and rendering today are becoming more complex and often contain different types of additives. This is a complication when analysing these mortars. Existing methods are useful for the analysis of pure lime mortars, cement mortars and lime cement mortars. As an example it can be mentioned that chemical analysis of mortars containing dolomite filler may give misleading results as the dolomite may not go into full solution in the acid used when dissolving the mortar. Similar problems apply to the analysis of historical mortars, which do contain a wide variety of additives in the mix. There is also a need for a more general method for the calculation of mix proportions based on the chemical analysis. The NT BUILD 436 method only provides a basis for the calculation of one mortar type. The problem is not the lack of methods, but the need for a more general approach to deal with the problem.

Work within RILEM TC 164 “Characterisation of historical mortars” has demonstrated the potential of a combination of chemical and microscopical methods. It is possible to use the strengths of both methods when interpreting the analytical results and to decide on the procedures used in the analytical work.

Thorborg von Konow at Tureida performed quantitative microscopical analysis through counting in fields. Thale Sofie Wester Plesser at SINTEF Byggforsk and Peter Nyman at SP performed chemical analysis using the NT BUILD 436 method. The analyses performed at SP included al hydraulic components. Both chemical and microscopical analysis were performed at TNO in Delft by Timo Nijland, Joe Larbi, and Rob van Hees. Jan Erik Lindqvist, SP, performed quantitative microscopical analysis.

The mortars were cast by Sten Johansson and the thin sections were prepared by Jan Winblad. Their care in this work is kindly acknowledged. The project has been financed through a grant from Nordic Innovation project number 04027 “Analys av hårdnat bruk med tillsatsmaterial” for the partner from the Nordic countries. The participation of TNO has been financed through their own research budget.

Sammanfattning

De metoder som finns för analys av mur och putsbruk är utvecklade för analys av enkla och väl definierade bruk. De bruk som används idag är i stor utsträckning hybridbruk som innehåller tillsatser av tillsatsmedel och tillsatsmaterial. Analys av dessa komplexa bruk ställer krav på analysrutiner som är mer flexibla. Syftet med detta projekt har varit att undersöka rutiner för karakterisering av komplexa bruk med hjälp av kemiska och mikroskopiska metoder. Målet har inte varit att ta fram en ny metod utan att formulera ett strategi för analys av komplex bruk. Förfaringssättet skall gå att tillämpa både när delmaterialen är kända och när delmaterialen från början inte är kända. För detta syfte tillverkades prismor av KC-bruk med dolomitfiller och av kalk cement slagg. Efter sex månaders härdning preparerades tunnslip för analys i ljusmikroskop och pulver för kemisk analys. För att bestämma mängden av olika delmaterial analyserades syralösliga komponenter kemiskt och delmaterialen kvantifierades i i ljusmikroskop med hjälp av punkträkning och räkning i fält. Från dessa resultat beräknades brukens blandningsproportioner. De kemiska metoderna gav en bedömning av blandningsproportionerna genom en generell algoritm baserad på den kända kemiska sammansättningen hos delmaterialen. Dessa kemiska analyser gav en god bedömning av brukets blandningsproportioner. Kvantitativa beräkningar baserade på resultaten från ljusmikroskopi utfördes dels enligt NT BUILD 370 och enligt metoder utvecklade på TNO. De mikroskopiska metoderna gav en god bedömning av kalkcementbruk och av slagbruk med låg slagghalt. Analys av bruk med hög slagghalt gav för låga beräknade ballast/bindemedelsförhållanden. De mikroskopiska metoderna var bra för att identifiera de olika delmaterialen i bruken. De erhållna resultaten visar att kombination av kemiska och mikroskopiska metoder kan ge en god bedömning av delmaterial och blandningsproportioner vid analys av dessa typer av komplexa bruk. Korrelationen mellan resultat från mikroskopisk analys och andelen av de olika delmaterialen enligt bruksrecepten var god. Resultaten från det genomförda projektet kan användas av laboratorier som gör analyser av bruk i samband med skadeutredningar och omputsning och omfogning av murverk i historiska monument. Fortsatt forskning inom detta område kan förmodligen formulera metoder även för bestämning av blandningsförhållanden hos slaggbruk och andra puzzolanbruk på samma sätt som det idag går att analysera kalkbruk och kalkcementbruk.

1 Introduction

1.1 Analytical strategy

A combination of chemical and microscopical analysis of hardened mortars can provide information on the type of binder, the hydraulic or pozzolanic properties and the mix proportions of the mortars. The aim of this project was to identify and quantify as far as possible the different materials in the mortar using chemical and microscopical methods. The quantitative data was then used to calculate the mix proportions. In a quantitative analysis of a complex mortar it is necessary to decide which components are to be quantified. For a quantification based on chemical analysis it is necessary to know the composition of the raw materials. In the present study the chemical analysis was performed using acid dissolution of the sample. Then the contribution of chemical components in the raw materials that are dissolved by the acid during the acid treatment of the sample must be determined. In the present project the aggregate and additives were also analysed. The contribution from TNO is also outlined in a separate presentation by Nijland et al 2005.

1.2 Chemical analysis

There are a large number of different methods used for the chemical analysis of acid-soluble components in hardened mortars. The NT BUILD 437 method is commonly applied in the Nordic countries. For the analysis of historical mortars the Florentine method (Vittori, C, Cereseto, A 1935) is widespread. Other useful methods are given in the recommendations by the RILEM TC-COM (Middendorf et al 2005). Table 1.1 gives a compilation of different methods for the chemical analysis of hardened mortars.

There are several problems involved in the acid dissolution of the sample. There may be difficulties in dissolving the paste in the mortar. Alvarez et al (1999) has made a comparison between using hot and cold hydrochloric acid with a concentration of 1:5. They concluded that the dissolution of the paste was incomplete when using hot acid. If there are fillers in the mortars these may contribute to the complexity. One example is that the attack by a weak acid on dolomite filler is slow and may not be complete.

There are also problems in keeping some of the components in solution. Silica may flocculate in a concentrated acid, e.g. 1:1, if the solution is allowed to stand for some time. With increasing acidity of the solution the size of the particles will also increase and a colourless silica gel may precipitate. However, if the analysis is done immediately after dissolution this method will also give reliable silica values (see figure 1.1). Alumina and iron are also difficult to keep in solution (Alvarez and references therein). The perchloric acid used in the NT BUILD method may be hazardous to handle in concentrated form. The perchlorate ion is larger and is less prone to form complexes than the chloride ion.

Table 1.1. Compilation of some methods for chemical analysis of acid-soluble components in hardened mortars.

Method	Acid	Concentration	Temp	Time	Milling	Reaction with dolomite	Solubility of Si in aggregate	Analysed acid-soluble components
1) NT BUILD 437	HClO ₄	1+9	Room T		90% <1.125 mm	Slowly soluble	Very little	CaO SiO ₂
2) Florentine method	HCl	5+1	-2 to 5	5 min	<0.1 mm	No attack (?)	Very little	
3) RILEM TC COM	HCl	Alt 1: 0.15 N Alt 2: 1+20	20 – 23 °C		aggregate >63 μ separated through sieving			SiO ₂ , Al ₂ O ₃ , CaO, MgO, SO ₃ , Na ₂ O, K ₂ O
4) Scancem Research	HCl	1:1 M HCl	4°C		Analyse fineness	Dolomite goes into solution	Comparable with the Nordtest method	
5) U84000150	HCl	1 N						
6) Alvarez	HCl	1:5	Warm/cold	30 min				
BS 4551:1970	HCl	1+9	50°C	5 h				
BS 4551:1980	HCl	1+9	22°C	20 min				

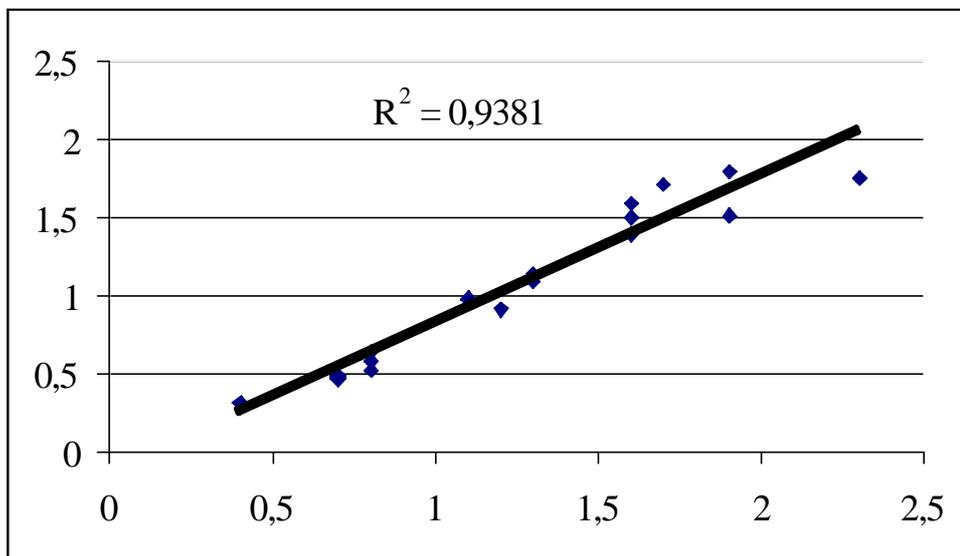


Figure 1.1. Comparison of analyses of acid-soluble silica using methods 1 and 4 in table 1. The analysed materials were mortars with a composition that compares with the dolomite filler containing mortar analysed in the present project. The results are given in weight percentage.

The type and concentration of acid, and the time and temperature of grinding of the sample before dissolution in acid, affect the degree of attack on the aggregate. Different kinds of aggregate have different solubility in acids. Minerals such as altered feldspars

and clay minerals may be partly soluble. Von Konow and Råman (1983) carried out a study of acid-soluble components in felsic sand from different producers in Finland using method 2 in table 1.1. The amount of acid-soluble SiO_2 was in the interval 0.08 – 0.45 with an average of 0.22 weight percentage. The corresponding value for CaO was 0.14 – 0.33 with an average of 0.21. Another approach is used in the RILEM TC COM (Middendorf 2005) method, where the aggregate in the carefully crushed sample is separated from the paste by sieving through a 63μ sieve before dissolution in acid.

Mortars may also contain pozzolanic materials. The definition of a pozzolan is that it reacts only in the presence of an alkaline “activator” and not as a hydraulic binder in the presence of only water. If a pozzolanic material is added to a mortar the reactive silica phases in the pozzolan will react with calcium in the fluids and form mainly CSH gel. There is no reaction when only water is added to the pozzolan. Blast furnace slag, as used in the present project, shows pozzolanic behaviour. The components of the CSH gel or other reaction products are acid-soluble. The reaction rate may however be slow and the mortar hardens before equilibrium is attained as it is hindered by the structure of the hardened paste.

1.3 Quantitative microscopical methods

There are, mainly, two different methods for quantitative assessment of mix proportions in mortars. They are the NT BUILD 370 (Sandström) and the quantitative methods used at TNO (Larbi and van Hees 2000a and b), which are applied in modified form in this project. The RILEM TC COM C1 method was developed from the NT BUILD 370 method (RILEM COM-C1 2001). The NT BUILD and TNO methods were developed independently but are fairly similar. In the procedure presented by Larbi and van Hees the density of the paste is analysed separately according to the RILEM recommendation CPC 11.3, and this is used as the input in the calculation. These two methods were developed mainly for the analysis of masonry and rendering mortars while the present project deals with more complex mortars. The limitations of the microscopical methods are the difficulty of obtaining representative samples, the difficulty of quantifying the hydraulicity of a hydraulic mortar and the fact that the method is very dependent on the experience of the operator. The advantage is the versatility of the microscopic method, as it can be used to identify and quantify a wide variety of additives in the mortars.

2 Methods applied in the present project

2.1 Preparation of the mortar prisms.

2.1.1 Materials used

The slaked lime used has a Ca(OH)_2 content of 98.3%. The dolomite filler used was Myanit A20 with a particle size less than 0.25 mm. The slag used was Merox Merit 5000. The cement used was ordinary Portland cement, OPC, CEM I. The compositions of the materials are given in table 2.1. The aggregate used was CEN-Normsand DIN EN 196-1.

Table 2.1 Chemical composition given in weight percentage and physical properties of the materials used. The compositions are obtained from 1 performed analyses, 2 product information, 3 Shaikh et al 1989.

	Cement ¹	Slaked lime	Slag ²	Dolomite ³
Silicon dioxide, % SiO_2	18.8	-	34	Approx. 1
Calcium oxide, % CaO	61.5	74.4	32	30.2
Magnesium oxide, % MgO	1.2	-	17.6	20.8
Sulphur, % SO_3	3.43	-	3.6	<0.01
Specific surface cm^2/g		-	5000	
Bulk density kg/m^3		-	1100	
Particle density g/cm^3	3.15	2.24	2.95	2.85
Glass content %		-	97-98	

2.1.2 Mixing procedure

The mortars were mixed and cast according to EN 1015-11. One exception was that the mortars were mixed for 5 minutes in the mixer. The cement, slag and dolomite were each homogenized before the aggregate was added to the mix. It was then homogenised manually before the mix was put in the mixer and water was added. The prisms were then cured at 20°C and 95% RH for five days and then 65% RH and 20°C for six months. The mix proportions are given in table 2.2 and the calculated chemical composition of the mortars in table 2.3.

Table 2.2. Mix proportions for the mortars used in the project given in grams of lime cement mortars with dolomite filler, upper part, and lime cement slag mortars, lower part. The flow test was performed according to the EN 1015-3 standard.

	Lime	Cement	Aggregate	Dolomite	Water	w/b	Flow test
LC15/85	45.0	255	1350	90	217.5	0.725	180
LC25/75	84.4	253	1350	51	220.8	0.654	174
LC35/65	94.5	176	1350	81	216.1	0.799	170
LC50/50	123	123	1350	37	220.5	0.896	174
LC65/35	146	78.8	1350	68	223.4	0.994	172
Cement type	Slag	Cement	Lime	Aggregate	Water	w/b	Flow test
CEM IIA-S	30.0	270	45.0	1350	215.3	0.624	-
CEM IIB-S	61.4	184	73.6	1350	212.0	0.665	181
CEM III/A	96.4	96.4	77.1	1350	205.7	0.762	183
CEM III/B	111	47.6	95.3	1350	203.7	0.802	-
CEM III/C	116	12.9	96.4	1350	200.1	0.888	175

Table 2.3. Chemical composition of the mortars given in weight percentage calculated from the composition of the raw material given in table 2.1 and the mix proportions given in table 2.2.

Sample	CaO	SiO ₂	MgO
LC 15/85	11.95	2.68	1.20
LC 25/75	12.77	2.62	0.74
LC 35/65	11.40	1.90	1.07
LC 50/50	10.43	1.37	0.54
LC65/35	10.37	0.90	0.87
CEM II A-S	11.82	3.45	0.49
CEM II B-S	10.85	3.21	0.77
CEM III/A	8.85	3.06	1.11
CEM III/B	8.26	2.85	1.26
CEM III C	7.26	2.61	1.31

2.2 Chemical Analysis of the mortars

The acid-soluble components in the samples were analysed at three different laboratories: SP and NBI, where the Nordtest method NT BUILD 437 was used, and TNO, using the method described below. In the NT BUILD 437 method the sample is ground to a powder and then moistened with absolute ethanol.

At NBI the following procedure, based on NT BUILD 437, is used:

- 4 g sample dried at 105°C for 2 hours
- Weigh sample
- Add 3 ml absolute ethanol
- Add 150 ml deionised water
- Add 10 ml perchloric acid, HClO₄
- Let mixture stir for 10 minutes
- Stop stirring, let stand overnight
- Filter mixture. Wash solid with deionised water.
- Dry solid at 105°C. Weigh solid
- Dilute filtrate to 250 ml

Analyse Si, Ca and Mg content in filtrate using flame atomic absorption spectroscopy (FAAS). The result is calculated as follows:

$$\% \text{ metal} = \frac{C_{\text{metal}} \cdot \text{Dil} \cdot 0.25}{W_{\text{sample}} \cdot 1000} \cdot 100\%$$

C_{metal} = metal concentration [mg/L]

Dil = dilution

W_{sample} = weight sample [g]

Table 2.4. Amount of dissolved dolomite given in weight percentage obtained using different acid solutions and temperatures.

No.	Solution				Method	Soluble dolomite
	ml		ml			
1	10	HCl	90	Water	Warm	97.9
2	25	HCl	75	Water	Warm	95.3
3	35	HCl	65	Water	Warm	89.0
4	45	HCl	55	Water	Warm	97.4
5	25	HCl	25	Water	Cold	88.1
6	25	HNO ₃	25	Water	Warm	88.7

To make sure that the dolomite used as filler was dissolved, several solutions were tested at TNO. The method used at TNO for dissolution of the samples was based on the dissolution test as follows. Given the results in table 2.4 the following procedure was used to dissolve the samples:

- 2 g sample was added to 10 ml concentrated HCl (37%) + 90 ml demineralised water
- the solution was heated to boiling
- the solution was placed in a container with water at 99°C for 15 minutes
- the filtrate was used for chemical analysis

At TNO analysis of Ca, Mg, and Si were obtained by atomic absorption spectrometry (AAS), analysis of sulphate by precipitation as BaSO₄ and analysis using flame photospectrometry. After dissolution of the sample, SiO₂ was analyzed using atomic absorption spectrometry (AAS). Two standard solutions of 100 and 200 ppm SiO₂ and a blank were used. Standards were prepared from an AAS-grade standard solution of 1000 pp SiO₂ from Acros Organics. For dilution, a solution of acidified Cs-La-chloride buffer was used. For AAS analysis, a 5 cm burner was used, together with a N₂O - acetylene mixture. At SP the concentrations of Ca, Mg, Si, Al, and Fe were analysed using ICP-OES. Total sulphur was determined using a Leybold induction furnace.

2.3 Microscopical analysis of mortars

The mortars were analysed in an optical microscope using thin sections measuring 34*44 mm². Point counting was used with 40x magnification at SP, and both laboratories used plain light. The methods used for calculating mix proportions are described further in the section calculation of mix proportions. SP used a modified version of the NT BUILD 370 and RILEM COM-C1 2001 method. The calculations used at TNO are based on Larbi and van Hees 2000a and b). Binder contents were also calculated using a modified version of the

procedure outlined by Larbi & Van Hees (2000a and b). In the presently used, modified, version, the amount of filler is also corrected for.

$$\text{Binder (wt.\%)} = (\text{Apparent density}_{\text{Mortar}} (\text{g cm}^{-3}) - \text{Aggregate (vol\%)} * \text{Density}_{\text{Agg}} - \text{Filler (vol\%)} * \text{Density}_{\text{Fil}}) / 1.25$$

Counting in fields was applied at Tureida. Each field had an area of 3.14 mm² and 50 fields were counted. The counting was performed in plain light and polarised light using the lambda plate. During the recalculation from number of grains to area percentage, an average area of 0.045 mm² was assumed for cement clinker, 0.03 mm² for dolomite and 0.0175 mm² for slag. The area percentage for the cement and slag grains obtained by calculation was recalculated as percentage of the binder, and that for the dolomite as percentage of the total mix. Tests with counting in fields were also performed at SP with good results, although the results were not used for further calculations.

In the mortars with dolomite filler the volume proportions of aggregate, binder paste, unreacted cement clinker and carbonate filler were determined through point counting on thin sections. In the slag mortars the volume proportion of unreacted glass was also determined.

3 Results

3.1 Chemical analysis

The amount of acid-soluble components in the prisms were analysed with slightly different methods at three laboratories. Results of the chemical analyses performed at the different laboratories are given in tables 3.1, 3.2 and 3.3. The raw materials used for casting the prisms were to some extent analysed using the same methods as used for analysis of the prisms, see tables 3.4. The relationships between mix proportions and analytical results are illustrated in figures 3.1, 3.2 and 3.3.

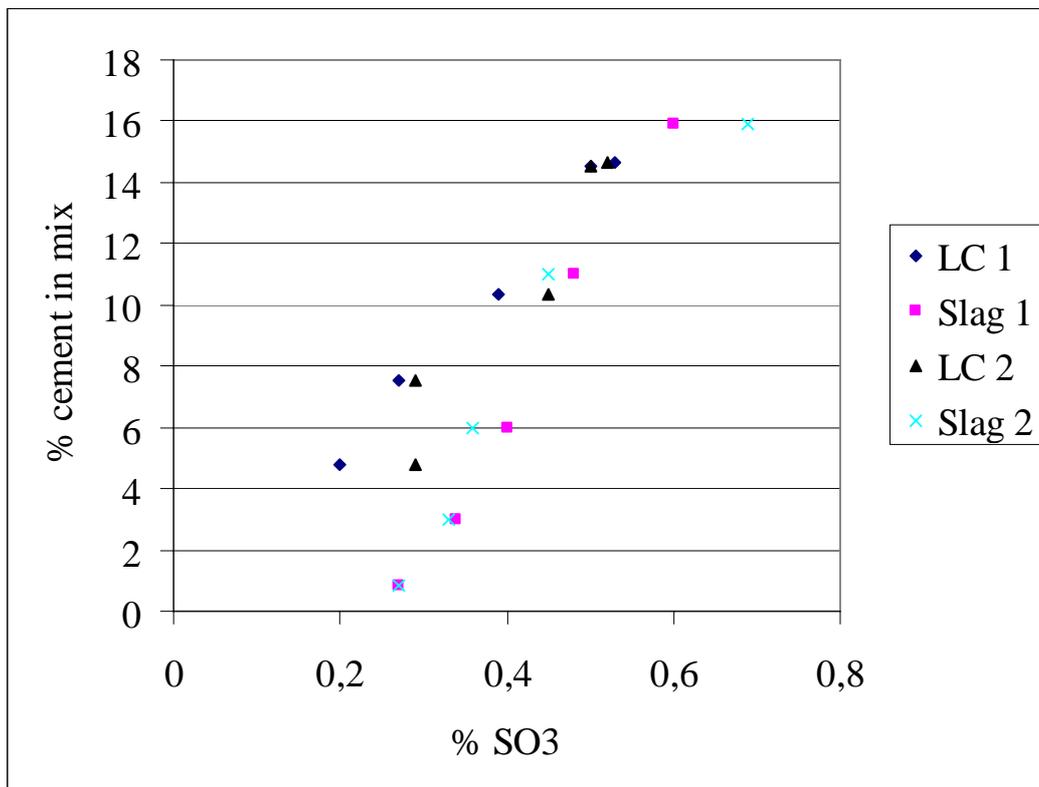


Figure 3.1. The relationship between weight percentage SO₃ analysed at two laboratories and the amount of cement in both lime cement mortars and lime cement slag mortars. The presence of sulphur (see table 3.4) also in other materials than the cement disturbs the relationship especially at low cement contents. As the sulphur content is different in the materials used the relationship will be slightly different for the two mortar types.

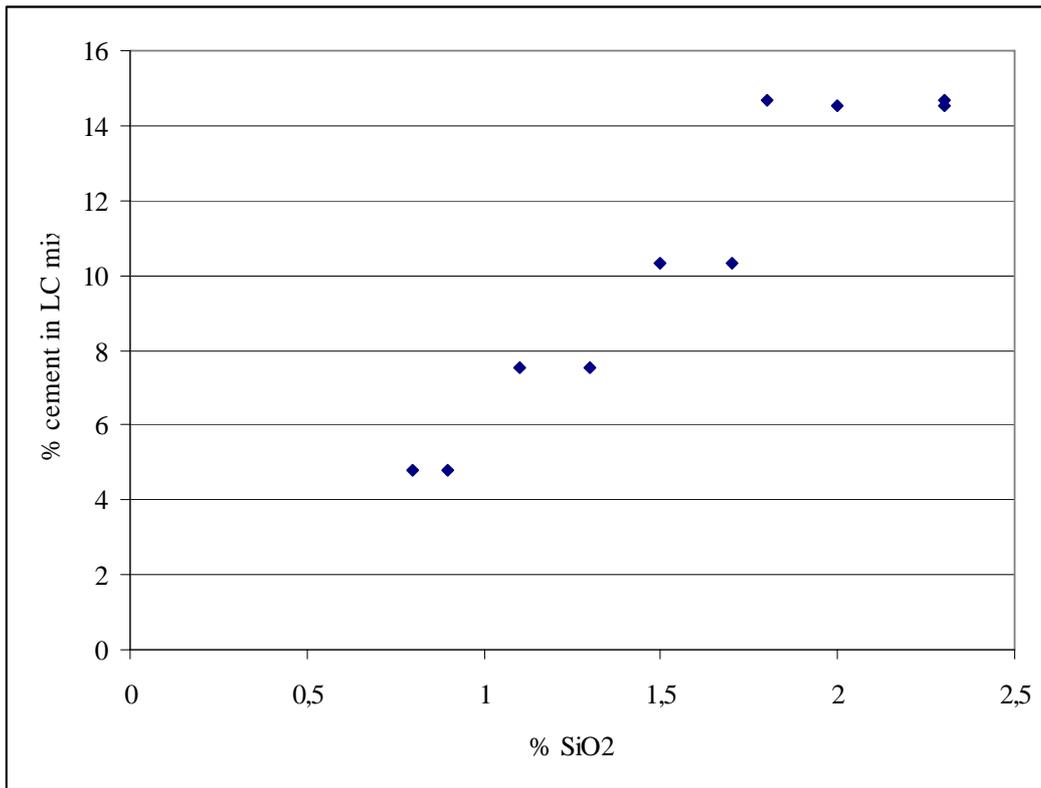


Figure 3.2. The relationship between weight percentage SiO₂ analysed at two laboratories and the amount of cement in the lime cement mix.

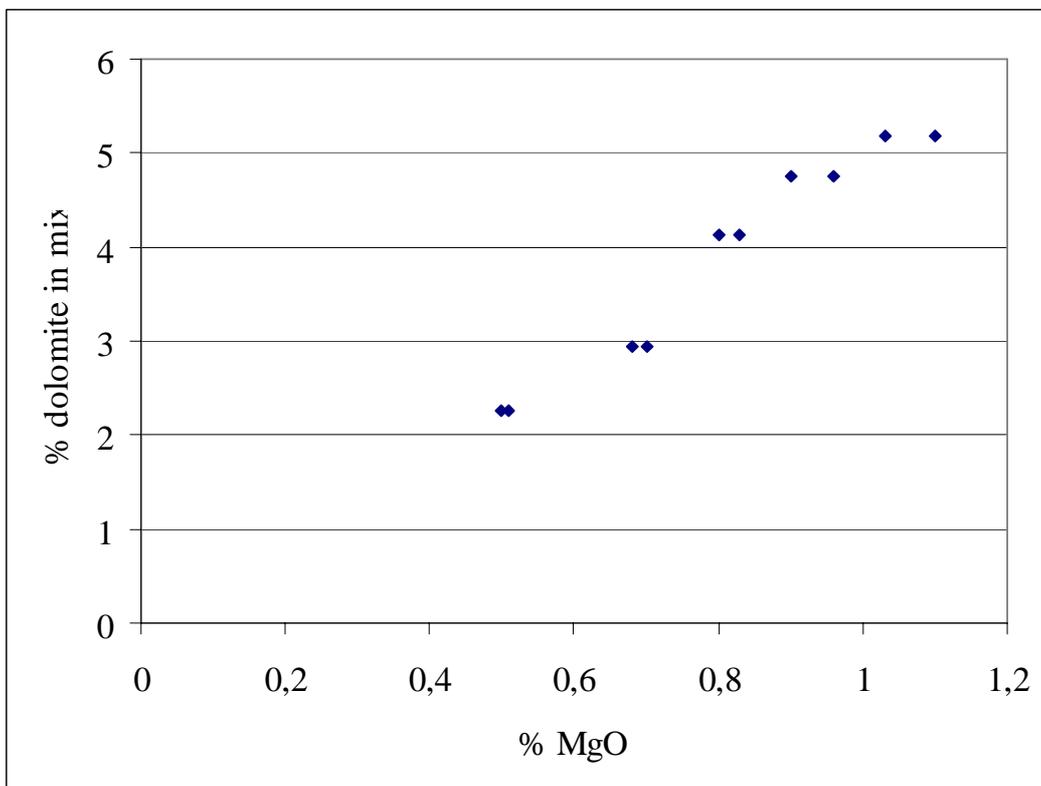


Figure 3.3. The relationship between weight percentage MgO analysed at three laboratories and the amount of dolomite in the lime cement mix.

Table 3.1. Results of the chemical analyses performed at SP. The results are given in weight percentage, Fe and Al are given as metals and not oxides.

Sample	SO ₃	CaO	SiO ₂	MgO	Fe	Al
LC 15/85	0.53	10.6	2.3	0.7	0.3	0.3
LC 25/75	0.5	11.6	2.3	0.4	0.3	0.3
LC 35/65	0.39	10	1.7	0.6	0.3	0.2
LC 50/50	0.27	9.1	1.1	0.4	0.2	0.1
LC65/35	0.2	9.3	0.8	0.7	0.2	0.1
CEM II A-S	0.6	11.1	3.1	0.4	0.3	0.4
CEM II B-S	0.48	9.6	2.8	0.6	0.2	0.4
CEM III/A	0.4	7.9	2.8	0.9	0.1	0.4
CEM III/B	0.34	7.4	2.6	1	0.1	0.4
CEM III C	0.27	6.6	2.4	1.1	0.1	0.4

Table 3.2. Results given in weight percentage from the chemical analyses performed at TNO.

Sample	SO ₃	CaO	SiO ₂	MgO
LC 15/85	0.52	11.25	3.8	1.03
LC 25/75	0.5	12.15	4.29	0.68
LC 35/65	0.45	10.86	3.24	0.96
LC 50/50	0.29	9.54	2.66	0.51
LC65/35	0.29	10.07	2.07	0.83
CEM II A-S	0.69	10.91	4.78	0.45
CEM II B-S	0.45	9.85	4.45	0.66
CEM III/A	0.36	9.38	4.83	0.95
CEM III/B	0.33	7.54	4.69	1.06
CEM III C	0.27	6.76	4.7	1.11

Table 3.3. Results given in weight percentage of the chemical analyses performed at NBI.

Sample	CaO	SiO ₂	MgO
LC 15/85	10.1	1.8	1.1
LC 25/75	11.15	2	0.7
LC 35/65	9.15	1.5	0.9
LC 50/50	8.7	1.3	0.5
LC65/35	8.9	0.9	0.8
CEM II A-S	10.15	2.7	0.4
CEM II B-S	8.7	2.5	0.6
CEM III/A	7.1	2.7	0.9
CEM III/B	6	1.2	1
CEM III C	5.85	2.2	1.1

Table 3.4. Analysis of the materials used when casting the mortar prisms. Dolomite has been analysed by three laboratories and the other materials at two laboratories.

	CaO	SiO ₂	MgO	SO ₃	Fe	Al
Dolomite	26.6	<0.1	16.6	<0.2	3	<0.02
Dolomite	32.82		18.52	0.25		
Dolomite	30	0	17.9			
Aggregate	0.58		0.03	0.28		
Aggregate	0	0	0			
Slag	39.08		16.43	0.14		
Slag	27	22.7	14.1			
Cement	61.63		1.08	3.64		
Cement	56.15	11.7	0.9			
Lime	80.3		0.61	0.24		
Lime	73.15	0.3	0.6			

3.2 Microscopical analysis

The results from point counting on LC mortars at SP are given in table 3.5 and for lime cement slag mortars in table 3.6. The results from point counting performed at TNO are given in table 3.7. The analysis performed by counting fields at Tureida is given in tables 3.8 and 3.9. The relationships between quantitative results and mix proportions are illustrated in figures 3.4 and 3.5.

For the determination of unhydrated or partly hydrated cement, as well as for the determination of dolomite filler and slag, a high number of counted points is needed in order to get acceptable precision in the analysis. The high number of counted points does not increase the statistical precision of the aggregate analysis as each large aggregate grain is counted several times.

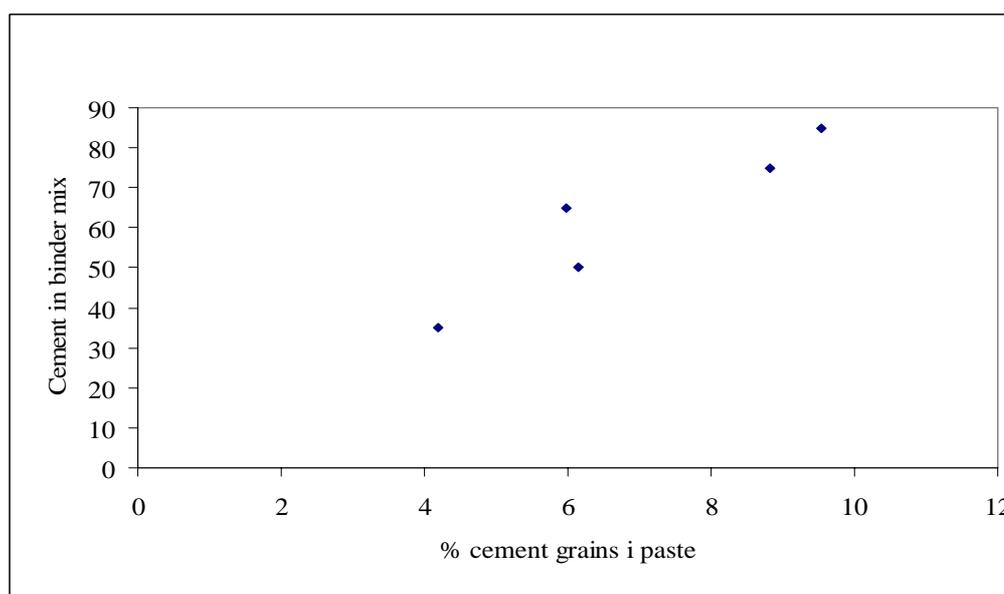


Figure 3.4. The analysed amount of unhydrated cement given in volume percentage in the sample (table 3.5) versus the weight percentage of cement in the mix for the lime cement mortars (table 2.2).

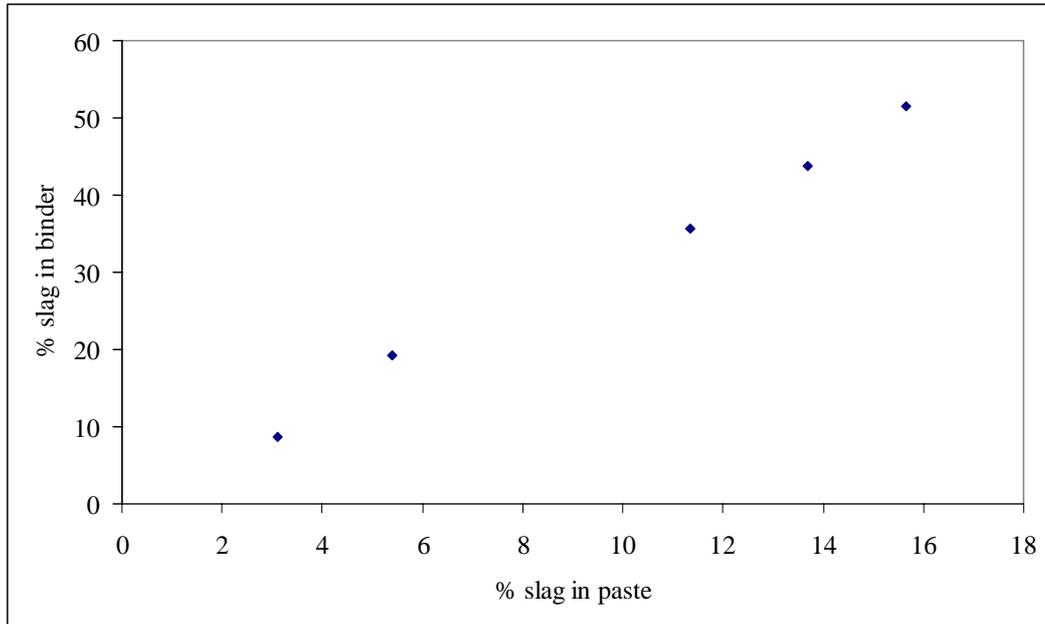


Figure 3.5. The analysed amount of unreacted slag given in volume percentage in the sample (table 3.6) versus the weight percentage of slag in the mix (table 2.2). The correlation is slightly better than the precision of the method which may give an overly optimistic view.

Table 3.5. Results from point counting on lime cement performed at SP. The upper part gives the number of points. The lower part gives the volume percentage in the analysed mortar based on the point counting. The precision given for cement and dolomite is calculated according to van der Plas and Tobi (1965).

Sample	LC15/85	LC25/75	LC35/65	LC50/50	LC65/35
Air	113	153	284	411	167
Aggregate	1593	1951	2717	1898	1557
Paste	919	1178	1511	1128	890
Cement	97	114	96	74	39
Limestone filler	70	76	148	44	64
Total	2792	3472	4756	3555	2717
Air	4.0	4.4	6.0	11.6	6.1
Aggregate	57.1	56.2	57.1	53.4	57.3
Paste	32.9	33.9	31.8	31.7	32.8
Cement	3.5 (± 0.7)	3.3 (± 0.6)	2.0 (± 0.4)	2.1 (± 0.5)	1.4 (± 0.4)
Limestone filler	2.5 (± 0.6)	2.2 (± 0.5)	3.1 (± 0.5)	1.2 (± 0.4)	2.4 (± 0.6)
Total	100	100	100	100	100

Table 3.6. Results from point counting on slag cement performed at SP. The upper part gives the number of points. The lower part gives the volume percentage in the analysed mortar based on the point counting. The precision given for cement and slag is calculated according to van der Plas and Tobi (1965).

Sample	CEMII A-S	CEMIIB-S	CEMIII/A	CEMIII/B	CEM III C
Air	200	213	344	374	340
Aggregate	1984	2042	2049	1853	2454
Paste	1143	1109	1033	1020	1197
Cement	104	82	36	20	14
Slagg	40	68	137	165	225
Total	3471	3514	3599	3432	4230
Air	5.8	6.1	9.6	10.9	8.0
Aggregate	57.2	58.1	56.9	54.0	58.0
Paste	32.9	31.6	28.7	29.7	28.3
Cement	3.0(± 0.6)	2.3(± 0.5)	1.0(± 0.3)	0.6(± 0.3)	0.3(± 0.2)
Slagg	1.2(± 0.4)	1.9(± 0.5)	3.8(± 0.6)	4.8(± 0.7)	5.3(± 0.7)
Total	100	100	100	100	100

Table 3.7. Microscopic identification of binder and filler (dolomite in case of LC samples or slag in case of CEM samples) performed at TNO, ¹ by point-counting, ² by estimating from a reference chart 'Diagrams representing various percentages of grains'.

Sample		Contents (vol.%)				
		Aggregate	Void	Binder	Filler ¹	Filler ²
LC15/85		64.2	4.9	27.7	3.2	7 – 10
LC25/75		57.4	4.9	31.3	6.4	5 – 7
LC35/65		53.5	9.8	32.3	4.4	5 – 7
LC50/50		56.4	9.4	30.0	4.2	3 – 5
LC65/35		57.6	5.2	29.3	7.9	3 – 5
LC65/35	duplo	58.8	5.9	28.5	6.8	3 – 5
CEM II/A-S		56.2	7.0	32.0	4.8	2 – 3
CEM II/B-S		60.4	6.0	31.2	2.4	2 – 3
CEM III/A		62.8	7.3	27.1	2.8	10 – 15
CEM III/A	duplo	63.1	6.9	30.0	-	10 – 15
CEM III/B		61.8	7.2	25.9	5.1	10 – 20
CEM III/C		51.5	19.6	21.3	7.6	20 – 25
CEM III/C	duplo	62.2	8.7	20.2	8.9	20 – 25

Table 3.8. Analysis of the number of cement and dolomite grains counted as number of grains in fields performed at Tureida. A total area of 157 mm² was analysed. The area percentage is calculated using the assumed sizes of 0.045 mm² for cement clinker and 0.03 mm² for dolomite, see text above.

Sample	Cement	Dolomite	Cement area %	Dolomite area %
LC 65/35	334	439	15	13
LC 50/50	534	324	24	10
LC 35/65	773	803	35	24
LC 25/75	870	268	39	8
LC 15/85	861	280	39	8

Table 3.9. Analysis of the number of cement and slag grains counted as number of grains in fields performed at Tureida. A total area of 157 mm² was analysed. The area percentage is calculated using the assumed sizes of 0.045 mm² for cement clinker and 0.0175 mm² for slag, see text above.

Sample	Cement	Slag	Cement area %	Slag area %
CEM II A-S	842	300	91	13
CEM II B-S	478	260	65	14
CEM III/A	302	403	32	17
CEM IIIC	100	932	10	37

4 Calculation of mix proportions

4.1 Chemical analysis

The calculation of the mix proportions from the analytical results can generally be seen as a system of equations with a number of unknowns. It gives one equation for each analysed component. The relationship between the analytical results and the calculated composition is illustrated in figure 4.1. It can be seen in the diagram that the relationship is different for the different mortar types.

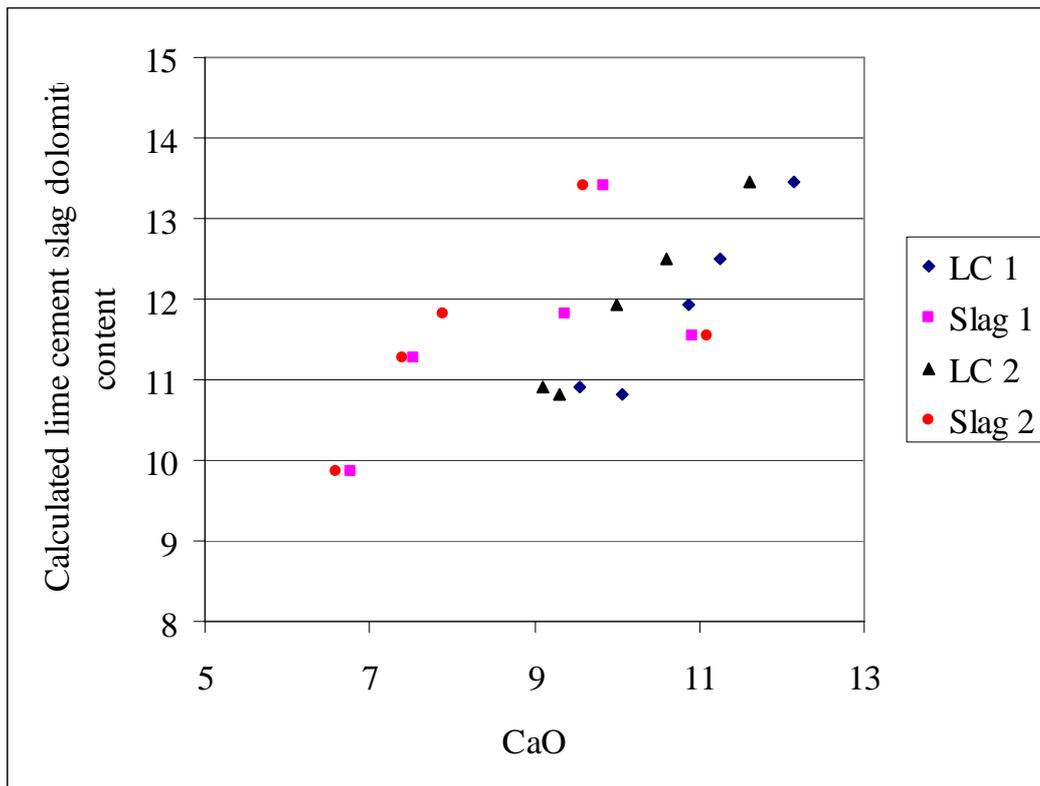


Figure 4.1. Shows the relationship for analysed weight percentage CaO and the amount of lime, cement, dolomite and slag respectively according to the formulas for CaO for LC mortars $61.5 \cdot X_{\text{Cement}} + 74.4 \cdot X_{\text{Lime}} + 30.2 \cdot X_{\text{Dolomite}}$ and $61.5 \cdot X_{\text{Cement}} + 74.4 \cdot X_{\text{Lime}} + 32 \cdot X_{\text{Slag}}$.

For the lime cement mortars this gives the following equations:

- 1) $SO_3_{\text{Analysis}} = SO_3 \cdot X_{\text{Cement}}$
- 2) $CaO_{\text{Analysis}} = CaO \cdot X_{\text{Cement}} + CaO \cdot X_{\text{Lime}} + CaO \cdot X_{\text{Dolomite}}$
- 3) $SiO_2_{\text{Analysis}} = SiO_2 \cdot X_{\text{Cement}} + SiO_2 \cdot X_{\text{Dolomite}}$
- 4) $MgO_{\text{Analysis}} = MgO \cdot X_{\text{Dolomite}} + MgO \cdot X_{\text{Cement}}$

And for the slag lime cement mortars this gives the following equations:

$$SO_3_{\text{Analysis}} = SO_3 \cdot X_{\text{Cement}} + SO_3 \cdot X_{\text{Slag}}$$

$$CaO_{\text{Analysis}} = CaO \cdot X_{\text{Cement}} + CaO \cdot X_{\text{Lime}} + CaO \cdot X_{\text{Slag}}$$

$$SiO_2_{\text{Analysis}} = SiO_2 \cdot X_{\text{Cement}} + SiO_2 \cdot X_{\text{Slag}}$$

$$MgO_{\text{Analysis}} = MgO \cdot X_{\text{Slag}} + MgO \cdot X_{\text{Cement}}$$

As an example the calculation of the mix proportion for LC15/85 can be used. The calculation here is based on the analytical results in table 3.1 and the chemical composition of the lime, cement and dolomite given in table 2.1. It gives the following equations

$$1 \text{ SO}_3) 0.53 = 3.43 * X_{\text{Cement}}$$

$$2 \text{ CaO}) 10.6 = 61.5 * X_{\text{Cement}} + 74.4 * X_{\text{Lime}} + 30.2 * X_{\text{Dolomite}}$$

$$3 \text{ SiO}_2) 2.3 = 18.8 * X_{\text{Cement}} + 1 * X_{\text{Dolomite}}$$

$$4 \text{ MgO}) 0.7 = 20.8 * X_{\text{Dolomite}} + 1.2 * X_{\text{Cement}}$$

$$X_{\text{Cement}} = \frac{0.53}{3.43}$$

$$X_{\text{Cement}} = 0.154$$

Which can be substituted into equation 4

$$20.8 * X_{\text{Dolomite}} = 0.7 - 1.2 * 0.154$$

$$X_{\text{Dolomite}} = 0.024$$

Substituted into equation 2:

$$74.4 * X_{\text{Lime}} = 10.6 - 61.5 * 0.154 - 30.2 * 0.024$$

$$X_{\text{Lime}} = 0.0054$$

The remaining equation can, in this case, be used as a control. The calculated mix proportions are given in the tables 4.1 and 4.2. The values for $X_{\text{Cement}} + X_{\text{Lime}}$ have been recalculated as 100 to give weight mix proportions. This is then applied according to the formula:

$$\text{Mix Pr op Lime} = \frac{100 * X_{\text{Lime}}}{X_{\text{Cement}} + X_{\text{Lime}}}$$

Table 4.1. Calculated compositions given in weight proportions for LC mortars, based on the chemical analysis given in tables 3.1 and 3.2.

	LC15/85	LC25/75	LC35/65	LC50/50	LC65/35
L/C/A/D	LC15/85/450/30	LC25/75/400/15	LC35/65/500/30	LC50/50/550/15	LC65/35/600/30
Lime	6	18	14	37	38
Cement	94	82	86	63	62
Aggregate	496	446	528	627	611
Dolomite	25	14	25	15	26
Lime	3	18	22	39	53
Cement	97	82	78	61	47
Aggregate	513	460	574	658	690
Dolomite	16	6	15	11	25

Table 4.2. Calculated compositions given in weight proportions, based on the chemical analysis from two laboratories, for the lime cement slag mortars.

Type	CEM IIA-S	CEM II B-S	CEM III/A	CEM IIIB	CEM IIIC
L/C/S/A	14/86/10/429	29/71/24/524	44/56/56/778	67/33/78/945	88/12/106/1235
Lime	neg	27	54	56	79
Slag	6	24	49	62	82
Cement	100	73	46	44	20
Aggregate	426	615	748	1062	1348
Lime	5	18	30	49	78
Slag	7	19	41	56	81
Cement	95	82	70	51	22
Aggregate	474	614	886	1025	1389

4.2 Calculation of cementation index

For mortars with unknown binders the calculation of the hydraulicity provides a useful basis for classification. It is mainly used for natural hydraulic limes. The most widely used is probably the cementation index given by Eckel (1922) and Boynton (1980):

$$CI = \frac{2.8 * SiO_2 + 1.1Al_2O_3 + 0.7Fe_2O_3}{CaO + 1.4MgO}$$

The mortars are then classified as feebly hydraulic 0.3 – 0.5, moderately hydraulic 0.5 – 0.7 and eminently hydraulic 0.7 – 1.1. An OPC has a CI of approximately 1.0. The system is intended for natural hydraulic limes but can be used to get an indication of the type of binder used when analysing an unknown mortar type. Calculated CI values are given in table 4.3.

Table 4.3. Calculated cementation index (CI) for the different mortars. The values given in table 3.1 are used.

Sample	LC15/85	LC25/75	LC35/65	LC50/50	LC65/35
CI Boynton	0.60	0.59	0.48	0.35	0.24
Sample	CEM II/A-S	CEM II/B-S	CEM III/A	CEM III/B	CEM III/C
CI Boynton	0.81	0.80	0.87	0.84	0.83

4.3 Mix proportions based on microscopical analysis

The mix proportions based on the results given in tables 3.5 and 3.6 have been calculated using the principles given in the NT BUILD 370 method and the results are given in table 4.4 for lime cement mortars and in table 4.5 for lime cement slag mortars.

Applying the NT BUILD 370 method for a lime cement mortar, the weight proportion of aggregate/binder (F) is calculated using the equation:

$$F = \frac{\alpha * \text{volume aggregate}}{\text{volume paste} - \text{volume unhydrated cement clinker}} - \beta$$

The term β varies between 0.5 and 1.5 depending on the minute suction of the substrate. This is a correction for the water content in the mortar, which affects the hydration and the porosity of the mortar. The term α depends on the density of paste, aggregate and the water content. It is about 2.2 for a cement mortar and about 3 for a lime mortar. For a cement mortar it is given by:

$$\alpha = \frac{\text{aggregate density}}{\text{density paste} * (1 - \text{water content})}$$

and is approximately:

$$\alpha = \frac{2.67}{1.75 * (1 - 0.3)}$$

this is then used in the calculation of F according to the equation:

$$F = \frac{\alpha * \text{volume aggregate}}{\text{volume paste}}$$

Table 4.4 Calculated weight mix proportions for lime mortars based on the NT BUILD 370 method and on the results from microscopical analyses given in table 3.5. The mix values are the weighted mix proportions when the prisms were cast, and the calculated values are based on the quantitative results from point counting.

LC 15/85	L	C	A	F
Mix	15	85	450	15
Calculated	37	63	452	22
LC 25/75	L	C	A	F
Mix	25	75	400	15
Calculated	41	59	433	19
LC 35/65	L	C	A	F
Mix	35	65	500	30
Calculated	55	45	491	29
LC 50/50	L	C	A	F
Mix	50	50	550	15
Calculated	54	46	455	12
LC 65/35	L	C	A	F
Mix	65	35	600	30
Calculated	64	36	486	22

Table 4.5. Calculated weight mix proportions for lime cement slag mortars based on the NT BUILD 370 method and on the results from microscopical analyses given in table 3.6. The mix values are the weighted mix proportions when the prisms were cast and the calculated values are based on the quantitative results from point counting.

CEM IIA/S	L	C	A	S
Mix	14.3	85.7	430	10
Calculated	43	57	459	10
CEM IIB/S	L	C	A	S
Mix	29	71	525	24
Calculated	51	49	499	18
CEM IIIA	L	C	A	S
Mix	45	55	780	55
Calculated	68	32	563	41
CEM IIIB	L	C	A	S
Mix	66	33	945	78
Calculated	75	25	520	51
CEM IIIC	L	C	A	S
Mix	88	12	1235	106
Calculated	79	21	598	59

Binder contents have also been calculated using a modified version of the procedure outlined by Larbi & Van Hees (2000ab). In this modified version, the amount of filler is also corrected for:

$$\text{Binder (wt.\%)} = (\text{Apparent density}_{\text{Mortar}} (\text{g cm}^{-3}) - \text{Aggregate (vol\%)} * \text{Density}_{\text{Agg}} - \text{Filler (vol\%)} * \text{Density}_{\text{Fil}}) / 1.25$$

Results are given in table 4.6, with binder contents depending on the filler content. The following data have been used: aggregate and filler contents from table 7, apparent densities from table 8, as well as the following densities of materials: quartz sand 2.65 g cm^{-3} , dolomite 2.87 g cm^{-3} , slag 2.90 g cm^{-3} .

The calculated results based on counting in fields are given in table 4.7. The area percentage for the cement and slag grains obtained by calculation was recalculated as percentage of the binder and that for the dolomite as percentage of the total mix.

Table 4.6. Calculated weight mix proportions based on the TNO method.

Sample		Calculated binder content (wt.%)			Cement + lime (kg)
		Filler ¹	Filler ²	Filler ²	
			Min	Max	
Lime – cement – dolomite					
LC15/85		31	22	15	300
LC25/75		29	32	28	337
LC35/65		44	42	38	290.5
LC50/50		32	34	30	246
LC65/35		25	36	32	224.8
LC65/35	duplo	28	36	32	224.8
Lime – cement – slag					
CEM II/A-S		36	43	41	315
CEM II/B-S		33	34	32	257.6
CEM III/A		25	8	neg.	170
CEM III/A	duplo	-	8	neg.	170
CEM III/B		24	12	neg.	142.6
CEM III/C		11	neg.	neg.	109.3
CEM III/C	duplo	8	neg.	neg.	109.3

Table 4.7. Calculated weight mix proportions based on counting in fields.

LC 15/85	L	C	F	CEM IIA/S	L	C	S
Mix	15	85	5	Mix	13	78	9
Calculated	37	78	5	Calculated	11	78	11
LC 25/75	L	C	F	CEM IIB/S	L	C	S
Mix	25	75	3	Mix	23	58	19
Calculated	41	80	5	Calculated	46	45	9
LC 35/65	L	C	F	CEM IIIA	L	C	S
Mix	35	65	5	Mix	29	36	36
Calculated	55	74	15	Calculated	56	29	15
LC 50/50	L	C	F	CEM IIIB	L	C	S
Mix	50	50	2				
Calculated	54	52	6			not calculated	
LC 65/35	L	C	F	CEM IIIC	L	C	S
Mix	65	35	4	Mix	43	6	51
Calculated	64	32	8	Calculated	55	10	35

5 Discussion

5.1 Chemical

Chemical analysis can provide a good assessment of the mix proportions in a mortar provided that the raw materials and their compositions are known. Otherwise it is recommended to start with a microscopic analysis using thin-section technique before doing the chemical analysis. The alternative is to calculate the cementation index and give an approximate estimate of the aggregate paste ratio. The crucial step in the analysis is the dissolution of the sample. If the raw materials are available a test of the yield in different acid solutions can give useful information when deciding on the method used.

The cement contents of both mortars show reasonable correlation with the SO_3 contents of the solution. Calculation of the cement content from the sulphate content assumes that all sulphate comes from the cement, which is not strictly true, see table 3.4. This decreases the reliability of the cement determinations especially at low cement contents. The analysis of acid-soluble SiO_2 , which can also be used for calculation of the cement content, shows a rather large discrepancy between the different laboratories.

5.2 Microscopical

Microscopical methods gives a good assessment for the lime cement mortars and for mortars with a low slag content, while the calculated aggregate-binder ratios and the filler contents are too low for the slag rich mortars. The quantitative results show however a clear correlation between analysed slag contents and the mix proportions. It is likely that the analysis of other pozzolanic materials would give similar results.

In the case of the slag, not all the particles in the gradation used can be seen and counted. Some of the very fine particles, especially those that are smaller than, for example $5 \mu\text{m}$, have to a large extent already reacted with the alkaline pore solution to form part of the cement paste. As the slag mortars in this investigation contain an excess of available calcium for slag to form calcium silica gel, the hydration of the slag may proceed very far and consume a large portion of slag. This reduces the amount of slag particles counted and consequently increases the amount of paste in the mortar. This may account for the difference in aggregate paste ratio calculated from the analytical results compared to the mix proportion. When a reference chart is used, the estimation is done randomly and is based on a range, which depends on the number of particles of slag that are seen in the field of view of the microscope. Since the distribution of the slag particles in the binder and mortar is not uniform and moreover only a small area of view is examined at a time (for example $1.4 \text{ mm} \times 0.9 \text{ mm}$), one may have to examine several such areas in order to obtain a reliable estimate, which is practically cumbersome.

There is a difference in which slag and cement grains are counted between the different laboratories. For cement and slag the operator has to decide how reacted cement grains shall be counted. The different operators must therefore establish their own calibration curves that are adapted to the interpretation they are using.

Other problems occur if the mortar has calcite filler rather than dolomite filler. It is then impossible to obtain the amount of calcite filler separated from the lime in the mortar through chemical analysis. In this case the microscopic method gives a more reliable result.

6 Recommendation

If the composition and raw materials of the samples are unknown it is recommended to start with a thin-section analysis in an optical microscope. The first step is then to identify the constituents in the mortar. These can then be quantified using point counting or counting in fields. The mix proportions can be assessed from these data using the methods outlined in part 4.3. The results from the present project show however that the interpretation of these data for mortar types where there is limited experience in quantitative assessment should be done with care.

Once the type of mortar and the raw materials have been identified it is possible to perform a chemical analysis. A first step is to decide on the method used for dissolution in acid. If the raw materials are known a test of the yield could be performed as described in section 2.2 above. The method used for the chemical analysis of the filtrates is not a crucial part of the procedure and different methods are applied at different laboratories. If the chemical composition of the raw materials are known, or a composition can be reliably assumed, a calculation of mix proportions based on the analytical results can be made using the method described in 4.1 above. If the type of mortar is not known a calculation of the cement index, CI, as described in section 4.2 above, can be used to characterise the binder. This can be combined with an approximate assessment of the binder-aggregate ratio.

The microscopical method gives information on the constituents in the mortar and the mix proportions but it gives limited information on hydraulic and pozzolanic properties of the binder. The chemical method can give information on mix proportions and also the on hydraulic and pozzolanic properties of the binder but it gives limited information on the type of binder and other raw materials.

A recommended approach can be as follows:

1. Mortar sample of unknown raw materials start at step 2 if the raw materials is know go to step 3.
2. For samples with unknown raw materials start with thin section analysis in order to identify the constituents.
3. Sample with known raw materials. For analysis using quantitative microscopical analysis on thin sections go to step 7. Chemical analysis of acid soluble components including CaO, SiO₂, if the hydraulic properties of the mortar are important include also Al₂O₃, Fe₂O₃ and MgO. This can be performed according to steps 4 to 6.
4. Select a suitable method for the acid solution of the sample. This can be performed according to NT BUILD 437. If there is a doubt whether the materials are fully soluble in the acid then it is possible to make a test of the yield in different acids, concentrations and temperatures.
5. For non hydraulic and non pozzolanic mortars analysis of CaO and SiO₂ together with loss on ignition is sufficient. For hydraulic mortars is it recommended to also include Al₂O₃, Fe₂O₃ and MgO in the analysis.
6. Calculation of results. For cement, lime cement and lime mortars can the NT BUILD 436 procedure be applied. For other types of mortars where the composition of the raw materials (*R*) is known can the amount of the different raw materials (*XR in mortar mix*)

be calculated using the following procedure. In this example with three raw materials contributing with acid soluble components:

$$1) SO_3 \text{ Analysis} = (SO_3 \text{ in } R1) * (XR1 \text{ in mortar mix}) + (SO_3 \text{ in } R2) * (XR2 \text{ in mortar mix}) + (SO_3 \text{ in } R3) * (XR3 \text{ in mortar mix})$$

$$2) CaO \text{ Analysis} = (CaO \text{ in } R1) * (XR1 \text{ in mortar mix}) + (CaO \text{ in } R2) * (XR2 \text{ in mortar mix}) + (CaO \text{ in } R3) * (XR3 \text{ in mortar mix})$$

$$3) SiO_2 \text{ Analysis} = (SiO_2 \text{ in } R1) * (XR1 \text{ in mortar mix}) + (SiO_2 \text{ in } R2) * (XR2 \text{ in mortar mix}) + (SiO_2 \text{ in } R3) * (XR3 \text{ in mortar mix})$$

7. Calculation of the Cement Index (CI), which can be used as a measure of the hydraulic and / or pozzolanic properties, can be done according to the following formula:

$$CI = \frac{2.8 * SiO_2 + 1.1 Al_2O_3 + 0.7 Fe_2O_3}{CaO + 1.4 MgO}$$

8. A quantitative microscopical analysis is performed on thin sections using point counting, counting in fields or line analysis. The results can be presented as volume parts in the hardened mortar but it may also be recalculated as mix proportions according to the steps 9 and 10.

9. For cement, lime cement and lime mortars can the calculation of the mix proportion be done according to NT BUILD 370 or Larbi and van Hees (2000a and b).

10. For mortars made from other types of raw materials can the mix proportions for non reactive raw materials with known particle density, such as fillers, be calculated using the NT BUILD 370. For reactive raw materials can the method be applied using an assumption concerning the reactivity. This assessment of mix proportions in mortars with pozzolanic and other reactive raw materials should be applied cautiously

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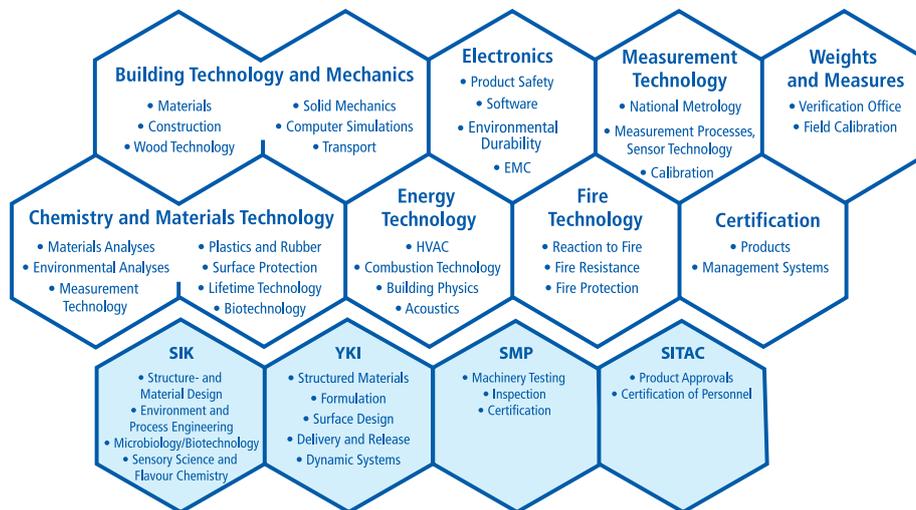
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SP Swedish National Testing and Research Institute

Box 857
 SE-501 15 BORÅS, SWEDEN
 Telephone: + 46 33 16 50 00, Telefax: +46 33 13 55 02
 E-mail: info@sp.se, Internet: www.sp.se

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