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THE DETERMINATION OF MOISTURE CONTENT OF THE AGGREGATES WITH MICROWAVE OVENS FOR THE USE OF CONCRETE PRODUCTION

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The target of this thesis was to validate the use of microwave ovens in determining the moisture content of aggregates. Parameters to be optimized are the mass of wet sample, total duration of the test, power setting of the micro wave oven and the sequence of heating intervals during the testing. Four standard aggregate sizes were studied: filler 0/1 mm, sand 0/6 mm, crushed aggregates 4/8 mm and 8/16 mm. The test program was divided in 4 main stages: 1) validation of the microwave oven method, 2) determination of optimized power and heating time, 3) determination of optimum wet sample mass and 4) final simplifying of the procedure.

In the theoretical part of this thesis the main components of concrete which are binders, aggregates, water and admixtures are introduced. In order to understand the importance of the water-cement ratio of the concrete, its various effects are introduced, as well as factors affecting the water-cement ratio. In amongst of the factors affecting the water-cement ratio are the hydration process of the cement, different sources of effective water in concrete and current methods for determining the quantity of water coming from these sources. Also the procedure for calculation of the water-cement ratio is explained.

In the empirical part of this thesis a new method for concrete production, the use of microwave ovens for determining the moisture content of aggregates is validated. Also the test procedure is determined. The test parameters which are optimized are the mass of wet sample, total duration of the test, power setting of the microwave oven and the sequence of heating periods during the testing.

The aim of this study is was to find a quick, reliable and accurate test method for the determination of moisture content of aggregates. After this study it is obvious that the determination of aggregate moisture with the microwave oven method suits well for the use of concrete production. The microwave oven method was noticed to be quick, accurate, reliable and easy and light to work with in comparison to other gravimetric methods. Determination of the moisture content of aggregates with microwave oven has been noticed to be very useful in the concrete production, and in the OL3 project (construction of 5th nuclear reactor in Finland, to Olkiluoto) this method is now used daily, also the parameters defined in this thesis are applied. With this microwave oven method new moisture contents of aggregates can be defined shortly after the change in the concrete workability is noticed which improves the reliability of concrete production with flawless quality.

BETONIN TUOTANNOSSA KÄYTETTÄVÄN KIVIAINEKSEN KOSTEUSPITOISUUDEN MÄÄRITTÄMINEN MIKROAALTOUUNIN AVULLA

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Asiasanat: betoni, kiviaines, kosteus, testaus

Tämän insinöörityön tarkoituksena oli osoittaa kiviainesten kosteuspitoisuuden määrittäminen mikroaaltouunin avulla käyttökelpoiseksi menetelmäksi. Optimoitavat muuttujat menetelmässä ovat kostean näytteen massa, testin kokonaiskesto, mikroaaltouunin teho ja sopivien kuumennusaikojen jaksotus. Työssä tutkittiin filleriä 0/1 mm, hiekkaa 0/6 mm sekä murskattuja kiviaineksia 4/8 mm ja 8/16 mm. Testiohjelma on jaettu neljään pääasialliseen vaiheeseen: 1) mikroaaltouunimenetelmän vahvistaminen, 2) optimitehon ja lämmitysajan määritys, 3) kostean näytteen optimimassan määritys ja 4) menetelmän yksinkertaistaminen.

Insinöörityön teoreettisessa osiossa esitellään betonin osa-aineet: sideaineet, kiviainekset, vesi ja lisäaineet. Betonin vesi-sementtisuhteen tärkeyden ymmärtämiseksi sen aiheuttamat moninaiset vaikutukset ja siihen vaikuttavat tekijät esitellään. Vesi-sementtisuhteeseen vaikuttavia tekijöitä ovat sementin hydrataatioprosessi, tehollisen veden lähteet betonissa sekä näistä lähteistä peräisin olevien veden määrien selvittämiseen käytetyt menetelmät tällä hetkellä. Myös menetelmä vesi-sementttisuhteen laskemiseksi selitetään.

Työn empiirisessä osiossa osoitetaan kiviainesten kosteuspitoisuuden määrittäminen mikroaaltouunin avulla käyttökelpoiseksi menetelmäksi betonintuotannossa. Työssä optimoidaan kostean näytteen massa, testin kestoaika, mikroaaltouunin teho ja sopivien kuumennusaikojen jaksotus.

Tämän työn tavoitteena oli löytää nopea, luotettava ja tarkka kiviainesten kosteuspitoisuuden määrittämismenetelmän. Tämä työ osoittaa kiviainesten kosteuspitoisuuden määrittämisen mikroaaltouunin avulla soveltuvan hyvin betonintuotannon tarpeisiin. Mikroaaltouunimenetelmä osoittautui nopeaksi, tarkaksi ja luotettavaksi menetelmäksi sekä helpoksi ja kevyeksi suorittaa verrattuna muihin gravimetrisiin menetelmiin. Kiviainesten kosteuspitoisuuden määrittämisen mikroaaltouunin avulla on havaittu olevan erittäin käytännöllinen betonintuotannossa, ja tämä menetelmä on nyt päivittäisessä käytössä OL3-projektissa, missä rakennetaan Suomen viidettä ydinreaktoria Olkiluotoon. Myös tässä työssä määritetyt testiparametrit on otettu käyttöön. Mikroaaltouunimenetelmän avulla kiviainesten kosteuspitoisuus on mahdollista määrittää lyhyen ajan sisällä sen jälkeen kun muutos betonin työstettävyydessä havaitaan, mikä parantaa laadullisesti virheettömän betonin tuotantovarmuutta.

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APPENDICES

1. INTRODUCTION

Concrete is the most used material in the construction industry and in its basic form concrete consists of water, cement and aggregates. As a construction material, concrete can be cast into almost any desired shape or form, and after hardening it can form a structural, load bearing element.

Water-cement ratio is a very important factor in concrete production, and it has crucial effects to both, fresh and hardened concrete properties. In Finland the requirements for concrete production are defined in Betoninormit BY50 and it is the concrete producers responsibility to follow these regulations. This is not always an easy task to do, especially when special concrete, such as self compacting concrete or concrete with high consistency needs to be produced. A difference in water amount as small as 5 kg/m^3 can cause tremendous effects to workability of fresh concrete. The main variation in water amount comes from the aggregates, and aggregate moisture contents may vary due to the fact that aggregates from a new delivery with different moisture content are used. Also during wintertime when the aggregates are heated in order to get warm concrete, the aggregate mass can locally differ in temperature and moisture content affecting the workability of the concrete.

The moisture content of the aggregates can be determined using gravimetric or volumetric methods According to the standardized method the aggregate moisture is determined using a ventilated oven, the duration of this test is approximately 20 hours. The method of gas burners has duration of about 1 hour, results tend to be over-estimated and there is an occupational safety aspect, working with open flames and gas. Also volumetric moisture measurement systems, probes that read the aggregates moisture content, exist. In practice their reliability and ease of use is not sufficient for concrete production and therefore the use of these methods is rare. Based on the information about the current situation a new method for aggregate moisture measurement needs to be developed. To be suitable for every-day-use in a concrete batching plant the method needs to be simple, easy to use and produce accurate results quickly.

This thesis and all required tests were realized in the OL3 project. In OL3 project the construction of 5th nuclear reactor in Finland is realized, the new nuclear reactor is built to Olkiluoto, Eurajoki. The total quantity of concrete to produce in the project is approximately 160000 m^3. Four standard aggregate sizes were to be studied: filler 0/1 mm, sand 0/6 mm, crushed aggregates 4/8 mm and 8/16 mm. The target of this thesis is to validate the use of microwave ovens in determining the moisture content of aggregates. Parameters to be optimized are the mass of wet sample, total duration of the test, power setting of the microwave oven and the sequence of heating intervals during the testing. The temperature of the sample during test is also to be followed. The test program was divided in 4 main stages: 1) validation of the microwave oven method, 2) determination of optimized power and heating time, 3) determination of optimum wet sample mass and 4) final simplifying of the procedure.

2. CONCRETE

Concrete is the most used material in the construction industry and in its basic form concrete is produced from mixing water, cement and aggregates. Other chemical or mineral materials can also be added to the concrete mix in order to change or attain specific physical properties in the fresh or hardened concrete. As a construction material, concrete can be cast into almost any desired shape or form, and after hardening it can form a structural, load bearing element.

In this chapter other components of concrete than aggregates will be introduced. Aggregates, being the main subject of this thesis will be dealt separately in their own chapter.

2.1 Water

According to the standard SFS-EN 1008 (2002, 4-5) water coming from the communal drinkwater supplies, or natural water which is suitable for drinking are suitable for concrete production. Water from marshlands or bogs or water polluted by industrial or residual waste cannot be used without prior testing. Marshland waters are often acidic and rich in humus; they can also contain chemicals which are harmful to concrete, such as sulphides and sulphates. Humus is very harmful to concrete as it can remarkably slow or even inhibit the setting. Water for concrete should also be free of sugars since they also have a retarding effect on fresh concrete. The amount of chlorides in concrete, and its constituents is greatly limited as chlorides accelerate the corrosion of steel reinforcement. Water should be free from oils, fats and detergents as they can inhibit the hydration process, weaken the bonding between cementstone and the aggregates or cause excessive air voids inside the concrete. (Suomen Betoniyhdistys, 2005a, 62-63.)

2.2 Binders

Binders consist mainly of Portland cement clinker in combination with blast-furnace slag, silica fume, pozzolana, fly ash, burnt shale and limestone. Other minerals are also used but only in very small amounts. (EN 197-1, 2000 + A1 2004, 13.) All of these minerals have their own characteristics which can have diverse effects on fresh and hardened concrete. Through the choice of binders the hardening of concrete can be controlled.

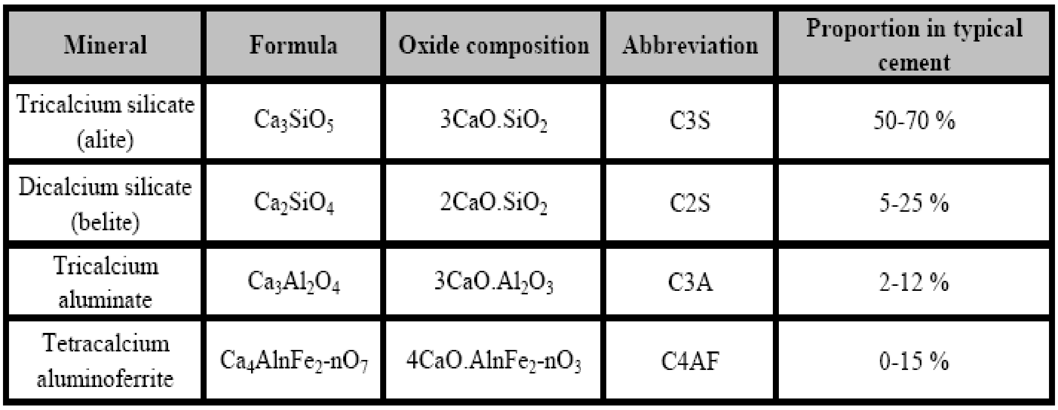
2.2.1 Cement

Cement is a finely ground, hydraulic binder, which reacts with water to form a hard and durable product. Together with water cement forms water-cement-paste which binds the other components together. The main component (80 %) in cement is Portland cement clinker, the rest 20 % is mainly clay. Portland cement clinker is produced by heating limestone (calcium carbonate $CaCO_3$) with small quantities of additives to 1400 °C in a calcination process. After calcination the clinker which at this point resembles a coarse aggregate is ground, together with a small amount of gypsum to a fine powder, called Portland cement. Gypsum is used to control the setting time of the cement. (Finnsementti, 1997, 11.) Portland cement is the most commonly used type of the cement. Apart from Portland cement, there are 4 other types of cement in modern day use. The European standard EN 197-1 (2000, 14) covers 5 categories, according to their chemical composition. These groups are

Portland cement, blended Portland cement, blast furnace cements, pozzolanic cements and composite cements, marked CEM I, II, III, IV and V, respectively.

Portland cement consists of four mineral groups: calcium, silicon, aluminium and iron oxides. An abbreviated nomenclature is commonly used to represent these groups and to simplify chemical formulae. These abbreviated nomenclature according to Barron (2010a, 1) are shown in the table 1.

Table 1. Components of Portland cement.



Mineral	Formula	Oxide composition	Abbreviation	Proportion in typical cement
Tricalcium silicate (alite)	Ca_3SiO_5	$3CaO.SiO_2$	C3S	50-70 %
Dicalcium silicate (belite)	Ca_2SiO_4	$2CaO.SiO_2$	C2S	5-25 %
Tricalcium aluminate	$Ca_3Al_2O_4$	$3CaO.Al_2O_3$	C3A	2-12 %
Tetracalcium aluminoferrite	$Ca_4AlnFe_2\text{-}nO_7$	$4CaO.AlnFe_2\text{-}nO_3$	C4AF	0-15 %

The composition of Portland cement can vary due to differences in material sources. Other minerals such as calcium or magnesium oxides may also be given to the Portland cement mixture, though only in small amounts.

Hydration, which means the reaction of water with the minerals, mentioned in the table 1 result in the setting and hardening of the cement stone. The process of hydration is not linear through time, it proceeds very slowly at first, allowing the water-cement paste, and resulting concrete to be placed and compacted before hardening begins. A curve that represents compressive strength of hardening concrete as a function of time is represented in the illustration 1. Even after long research the hydration process of cement, due to its complex tri-phasic system, is today still not fully understood.

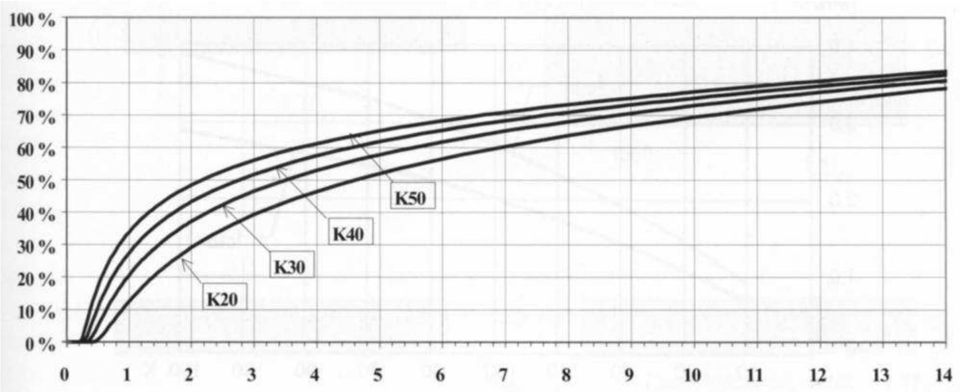


Illustration 1. Cement hydration is not linear through time (Suomen Betoniyhdistys, 2005a, 83).

Tricalcium silicate C3S provides the early age strength and resistance in concrete. Dicalcium silicate C2S reacts more slowly and contributes to the medium- and long-term strength development. The ratio of C3S and C2S in the cement clinker is used to determine the setting times of the cement paste. Setting times are faster when higher proportions of C3S are used and since C2S when reacting with water releases less energy than the reaction of C3S and water, higher proportions of C2S in the clinker will mean that less hydration heat will be released thus extending the setting time. Both C3S and C2S react with water to produce calcium hydroxide $Ca(OH)_2$ and calcium silicate hydroxide gel, C-S-H gel. C-S-H gel is the main product of the hydration process, and it gives concrete its strength. (Barron, 2010b, 1-2.)

The reactions of aluminate C3A and aluminoferrite C4AF, although these elements comprise usually total less the 25 % of the cement clinker, are very important. In comparison to the reaction of C3S, the hydration of C3A is very rapid and exothermic. If the hydration of C3A is not retarded the cement paste will set too quickly and the concrete cannot develop its expected strength. Gypsum is used to slow down the reaction of the C3A. With gypsum present C3A reacts together with water and gypsum to produce ettringite. The reaction of C4AF is very similar chemically to that of C3A though it takes place over a longer period of time. (Barron, A.R., 2010b, 3-4.)

The hydration of cement can be divided into four distinct phases. The illustration 2 represents these phases. In the square a (illustration 2) cement particles which have not reacted yet are surrounded by water. During the first few minutes the initial period of reactivity takes place and more reactive C3A and C4AF react with the gypsum and water to produce an amorphous gel on the surface of the cement grain, at the same time short rods of ettringite grow (square b, illustration 2). These reactions affect strongly the hydration of silicates. After this phase the cement hydration slows down for an induction period, which lasts approximately 3 hours (square c, illustration 2). After the induction period the process continues to acceleratory period which lasts approximately 20 hours. During this time the cement reacts with water to produce C-S-H gel and calcium hydroxide $Ca(OH)_2$ (square d, illustration 2). The C-S-H-gel is developed in two phases; during the first phase a layer of C-S-H is produced around the cement grain due to the reactions of C3S in the first 8 hours. In the latter phase C3A continues to react with the gypsum forming longer ettringite rods. This network of ettringite together with C-S-H gel forms a hydrating shell around the cement grain. The inner layer of C-S-H gel then forms inside this hydrating shell. After 1-3 days of hydration the reaction slows down and a decelerated period begins. The faster period is known as the setting phase and slower period as the hardening phase. (Barron, 2010b, 5.)

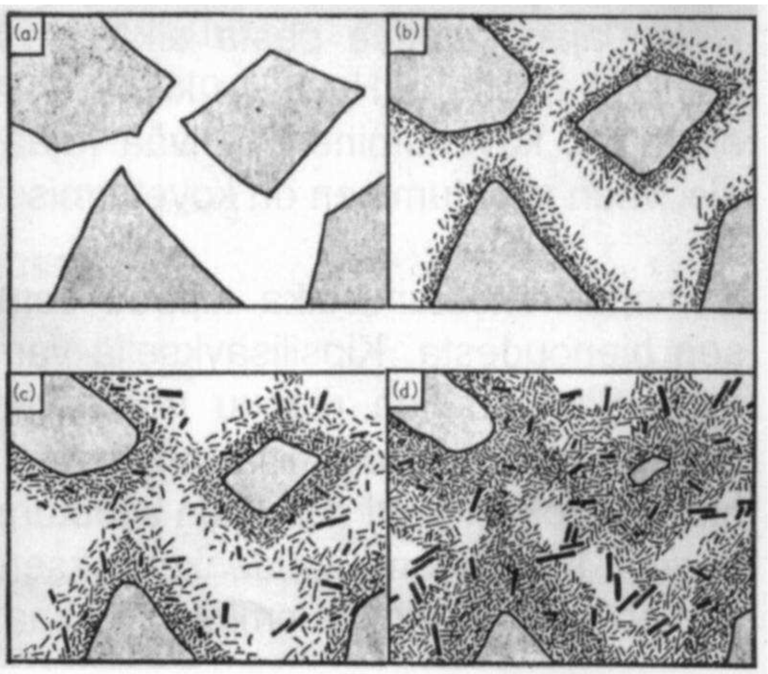


Illustration 2. Cement hydration can be divided into four periods (Suomen Betoniyhdistys, 2005a, 52).

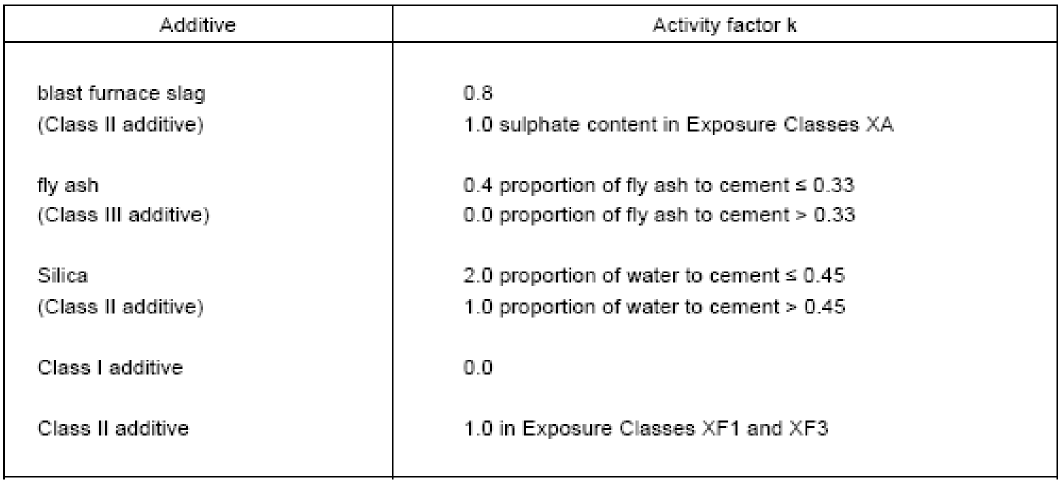
2.2.2 Additives

Most common types of additives used in Finland are fly ash, slag and silica. All of these are pozzolanic compounds which together with water and calcium hydroxide $Ca(OH)_2$ react to produce C-S-H gel. Calcium hydroxide is a by-product of the reaction between cement and water, so that the final product is similar to that produced by cement hydration. The hydration of cement must not be completed before the pozzolanic additives are able to begin reacting with the water and free calcium hydroxide. Pozzolanic additives do not participate in early strength development since reactions commences later and is much slower than that of cement. All of these mineral compounds can be used separately in concrete production or as additives in cement.

The reaction of slag is exothermic, but not as effective as that of cement. Through the use of slag the temperature development in hardening concrete can be controlled. The use of fly ash reduces the amount of air voids and the risk of fresh concrete segregation. Fly ash is generally used for self compacting concrete and when extremely fine surfaces are required. Silica is mainly used for the production of high-strength concretes. It always needs plasticizers as admixtures and demands a greater amount of water.

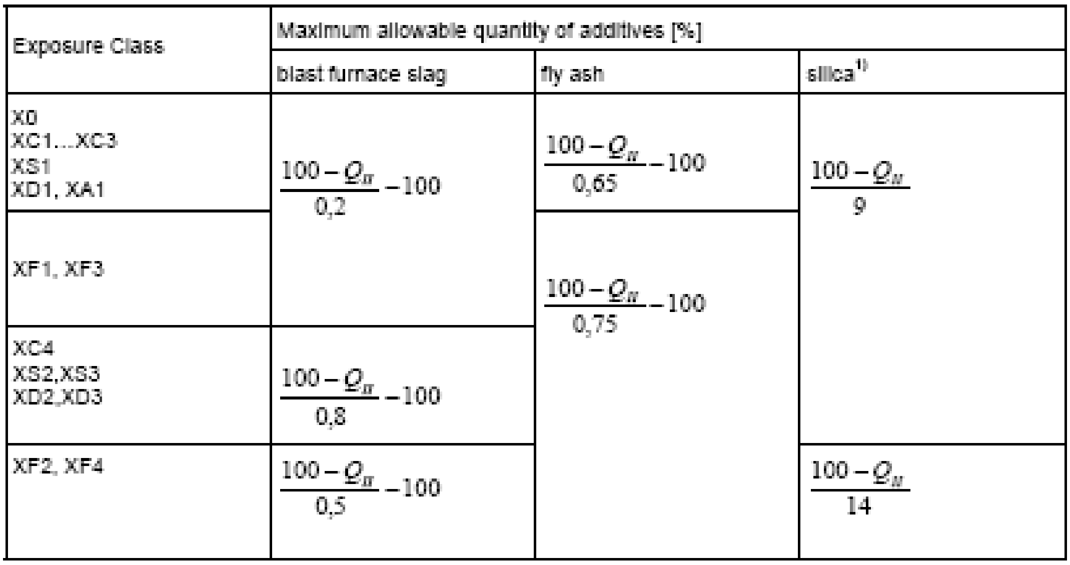
In the calculations of total binder content of the concrete, part of the fly ash, slag or silica content can be considered as a binder, thus making it possible to reduce the water-cement ratio or reduce the amount of cement needed. The amount of additive that can be considered as binder is calculated using the activity factor k. The activity factor depends on the classification of the additive and the concrete exposure class and they are represented in the table 2. For example when silica is used for normal-strength concrete with water-cement ratio less or equal to 0,45 its activity factor is 2. (Suomen Betoniyhdistys, 2005b, 100.) Therefore 1 kg of silica can be considered as 2 kg of cement. In fact various cements should have differing activity factors since additives can be an important component of the cement. Maximum allowable quantities of additives as percentages of cement mass, the requirement of BY50, are represented in the table 3 by concrete exposure classes. QII [%] indicates the additives contained by cement.

Table 2. Activity factor k of additives, when clinker activity is 1 (Suomen Betoniyhdistys, 2005b, 101).



Additive	Activity factor k
blast furnace slag (Class II additive)	0.8 1.0 sulphate content in Exposure Classes XA
fly ash (Class III additive)	0.4 proportion of fly ash to cement ≤ 0.33 0.0 proportion of fly ash to cement > 0.33
Silica (Class II additive)	2.0 proportion of water to cement ≤ 0.45 1.0 proportion of water to cement > 0.45
Class I additive	0.0
Class II additive	1.0 in Exposure Classes XF1 and XF3

Table 3. The maximum allowable quantity of additives [%] in concrete. Q_{II} indicates the additives [%] contained by cement. (Suomen Betoniyhdistys, 2005b, 101.)



Exposure Class	Maximum allowable quantity of additives [%]		
	blast furnace slag	fly ash	silica$^{1)}$
X0 XC1...XC3 XS1 XD1, XA1	$\frac{100-Q_{II}}{0,2}-100$	$\frac{100-Q_{II}}{0,65}-100$	$\frac{100-Q_{II}}{9}$
XF1, XF3		$\frac{100-Q_{II}}{0,75}-100$	
XC4 XS2,XS3 XD2,XD3	$\frac{100-Q_{II}}{0,8}-100$		
XF2, XF4	$\frac{100-Q_{II}}{0,5}-100$		$\frac{100-Q_{II}}{14}$

2.3 Admixtures

In concrete production various types of admixtures may be used, either singularly or in combination. Admixtures are used mainly for technical reasons which means that properties in the fresh or hardened concrete can be achieved which would be very difficult or impossible by using just cement. Examples of these are frost-resisting concrete or self-compacting concrete. Other aspect could be financial, which means that the use of admixture reduces other material cost. For example the use of plasticizer reduces the need of cement. Nevertheless the requirements for concrete and concrete production are too high that the use of admixtures for strictly financial reasons would be reasonable. The main types of admixtures are water reducing, air entraining, accelerating or retarding admixtures.

Traditional water reducing agents or plasticizers are usually lignosulfate based naphthalenes or melamines. Lignosulfates are by-products of the wood-processing industry, and due to impurities in the product, the dosage of plasticizers are limited. Typical dosage of plasticizers are < 1% of the cement mass, and with these dosages the amount of water reduction can be reduced up to 10 % without affecting the workability. (Suomen Betoniyhdistys, 2005a, 64-65.) This means that a lower water-cement ratio is present and higher compressive strengths can be achieved with the use of additional cement.

Superplasticizers or high-range water reducing agents (HRWR) are chemical products traditionally manufactured from melaminesulfonates or sulfonated naphtalenes. A new generation of polymer based superplasticizers such as polykarboxylethers (PCE) or polycarboxylates (PC) are also available. The dosage of superplasticizers is generally slightly higher than that of plasticizers, ranging between 1-3% for melaminesulfonates and up to 1,5% for other superplasticizers but a saving of up to 40 % water is achievable. (Suomen Betoniyhdistys, 2005a, 65.) Superplasticizers are used to produce self compacting or concrete with a very high workability which can be placed with little or no mechanical compaction at all. Especially when superplasticizers are used the correct proportion of fines has to be ensured to stabilize the fresh concrete to avoid segregation.

Air entrainment agents are surfactants, which form very small air voids, called protective pores, inside the concrete. These protective pores allow room for the water to expand in volume during freezing and thus reducing the internal pressure caused by freezing. Air entrained concrete can resist sub-zero temperatures and continuous freeze-thaw-cycles without cracking or other frost damages.

Retarders delay the start of the hardening phase so that concrete remains workable for a longer period of time. Retarders are mainly used in large pourings to avoid construction joints, or in situations where more time is needed to place and compact the concrete, for example difficult castings or where transportation takes longer. In warmer climates retarders are used to counteract the acceleration of setting caused by higher environmental temperatures and raised temperatures of hydration.

Accelerators increase the rate of early strength development, reduce the required curing and protection times and shorten the waiting time before finishing operations can be carried away. Accelerators are often used in small castings during winter, for larger castings it is more convenient to use heated concrete or heat treatment of concrete after placing or combination of the both. Chloride based accelerators were formerly the most common type used, but are rarely used nowadays due to their corrosive impact on steel reinforcement. Today, accelerators which are not chloride based are available but tend to be more expensive and less effective; they are not in such great use.

2.4 Aggregates

Aggregates form the main part in concrete, measured both in mass and in volume. Aggregates are divided into different size categories determined by the minimum and maximum grain size. In the concrete mix design the optimal particle size distribution curve is determined by proportioning the various aggregate size groups, fractions. The aim is to maximize the bulk density of the aggregates and minimize the voids between them, which otherwise must be filled with cement paste. Coarse aggregates due to their density and high compressive strength give the concrete its bearing strength, while fine aggregates are used to reduce the voids in the matrix, improve cohesion and compactness of fresh concrete, thus avoiding segregation and bleeding. In concrete production naturally graded, usually round, stones, crushed natural rock or artificial aggregates are used.

2.4.1 Naturally graded and crushed aggregates

Natural aggregates can be naturally occurring graded material or mechanically crushed from mineral soils or rock mass. Mineral soils consists of mainly inorganic stone types which over the years under the influence of water, weather and pressure have been reduced in size and formed in shape. In Finland repeated glacial periods have significantly affected the development of soils, and different types of soils have separated and formed layers. Because of this aggregate fractions suitable for use in concrete production are readily available. The most common soil type in Finland is moraine.

The consumption of aggregates in concrete production is very high, which means that sources need to be readily available. When aggregates need to be transported from further costs will raise and options may be limited. Especially in southern Finland it is difficult to find suitable naturally graded fine aggregates (Suomen Betoniyhdistys, 2005a, p.32). Good sources of suitable naturally graded aggregates are diminishing since removal of aggregates has begun to endanger the ground water sources; for this reason the possibility of underwater aggregate sources has been studied since 1980s (Uudenmaan ympäristökeskus, 2006).

In Finland mainly granite based aggregates are used and more and more often they are mechanically crushed. Rock masses in Finland are mainly hard enough for concrete production, but weathering of rock mass can be a problem in particular areas. Naturally graded and crushed aggregates have slightly different properties, mainly caused by different grain shape and surface textures. Using crushed aggregates the voids content and the combined aggregate friction are greater than when naturally graded aggregates with same grain size distribution are used. To achieve the same consistency with crushed aggregates higher percentage of finer part of combined aggregates and increase of water is usually needed. Increase of water leads to increase of cement to achieve the same water-cement ratio. On the other hand, the bonding between cement stone and crushed aggregates is stronger than the one between cement stone and naturally graded aggregates; also the impact resistance of crushed rock is greater. At least in theory this allows higher water-cement ratios to reach certain strength with crushed aggregates. However it has to be noted that shrinkage and creep of hardened concrete depend on the volume percentage of cement stone and water-cement ratio; these deformations increase with higher water-cement ratios. (Suomen Betoniyhdistys, 2005a, 46.)

2.4.2 Artificial aggregates

Artificial aggregates can also be used in concrete production. Utilization of waste material such as using recycled concrete or brick as aggregates is becoming general, and fly ash is used to replace fine particles. Also the by-product of the iron industry blast-furnace slag has been widely used in concrete production. Expanded clay aggregates are used to produce expanded clay light-weight concrete, also expanded shales, slates, perlites and slags are used. Properties of expanded clay aggregates differ from natural aggregates, grains absorb water and they crush easily for example during mixing. Also proportioning of expanded clay light-weight concrete is very different from normal concrete. Pelletized, air-cooled and granulated blast furnace slag can also be used, but the use is limited. (Suomen Betoniyhdistys, 2005a, 32.)

3. PROPERTIES OF AGGREGATES

Concrete consists mainly of aggregates, with 65-80 % of total volume. Therefore aggregates need to fulfil certain requirements, in general they have to be adequately hard and dense, inert towards cement and they should not weaken the durability of the concrete structure. They should not be weathered; and they also need to be clean from impurities, such as lumps of clay, oil, or snow and ice. (Suomen Betoniyhdistys, 2005a, 32.) According the BY50 aggregates used for concrete production have to meet the requirements of European standard EN-12620, and be CE marked. If CE mark is missing, aggregates need to be tested to testify the compliance with the standard. (Suomen Betoniyhdistys, 2005b, 97.) In general the properties of aggregates can be categorized in geometrical, mechanical, physical and chemical properties.

3.1 Geometrical properties

If aggregates were perfect spheres, the diameter would define their size distribution perfectly. However, aggregates differ in shape and surface properties. The size of aggregates is determined based on maximum dimension of the grain: whether one single grain of aggregate fits through a hole with certain size or it does not. The grain size distribution is determined by sieving, which can be done as dry or wet sieving. In the sieving process the percentages of certain grain sizes are measured based on their passing of sieves with known mesh size. The standard set of sieves consists of sieves with mesh sizes 0,125, 0,25, 0,5, 1, 2, 4, 8, 16, 31,5 and 63 mm. The grading curve and grading factor are obtained from sieving process. The percent fines (<0,063 mm) is determined with wash sieving, for that the standard set of sieves is complemented with a 0,063 mm sieve. An example of a grading curve is represented in the illustration 3.

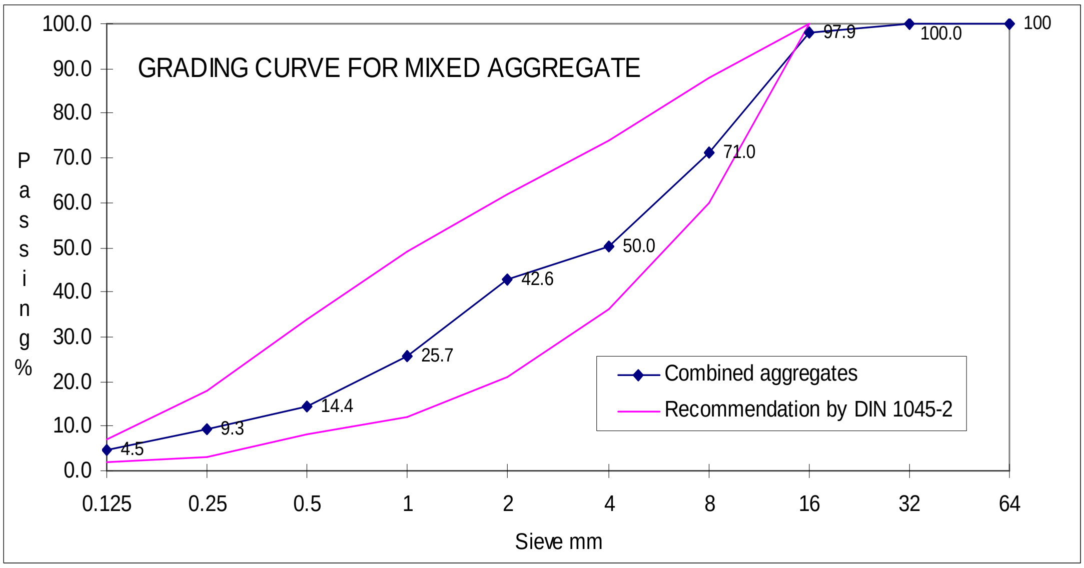


Illustration 3. An example of a grading curve for one concrete mix design used in OL3 project. Grading curve represents aggregate passings through standard set of sieves. Recommendation for grading curve is given in DIN 1045-2 (2001, 47).

Grain shape can be expressed as flakiness index or shape index. These indexes are used to express how much the shape differs from the sphere or cube. Grain shape can have an impact to strength and compactness of the concrete: sphere shaped aggregate grains can get closer to each other leaving only small empty spaces between them, concrete is dense and easy to compact. If aggregate grains are irregular in shape, even pin-like, grains can get stuck and they do not fill the space completely. As a result, concrete can be segregating or loose. Also more water-cement paste is required to fill the space between aggregates, and therefore concrete is more expensive to produce. Surface texture is described verbally: aggregates are glassy, rough, smooth or fairly rough or smooth. Surface texture can have an impact on concrete quality: with rough surfaces the bonding between cement stone and aggregate is stronger than with smooth surfaces. On the other hand, rough surfaced aggregates need a little bit more water than smooth, to be able to keep the same consistency, thus raising the water-cement ratio; therefore more cement is needed to achieve the required compressive strength.

3.2 Mechanical and physical properties

Specific gravity is the mass of unit volume of solid aggregate material compared to mass of one unit volume of water. Specific gravity does not include the void volume between the aggregate particles, and it does not have unit. The bulk density of aggregates gives the mass of bulk aggregate material in one unit of volume. Volume includes the volume of the aggregates and also the volume of voids between aggregate particles. The bulk density can be determined as loose bulk density or compacted bulk density. There are other ways of determine aggregate densities as well, depending on whether absorbed or only surface water is to be taken into account.

Water absorption of aggregates tells how much water totally dry aggregates will absorb. Absorbed water is inert, and it does not have an effect on concrete properties. On the other hand, if dry aggregates are used in concrete production, more water is needed, since aggregates absorb first some water inside the grains. Water absorption is very important factor in Finland, where freezing and freeze-thaw-cycles have to be taken into account. In general Finnish aggregates are frost-resistance and water absorption of normal, un-weathered aggregate is between 0,3-0,5 % (Suomen Betoniyhdistys, 2009, 23).

Aggregate moisture tells how much free surface water aggregates have. This water is able to affect the concrete, and it can have a remarkable impact to concrete quality. Moisture of aggregates, being the main topic of the thesis, will be introduced in the next chapter.

Properties such as strength, toughness, hardness, durability and soundness are mechanical properties. Those are influenced by other properties, such as surface texture, grain size distribution, mineral composition and weathering. In Finland mechanical properties of aggregates are relatively good, but especially in demanding jobsites also these have to be checked. For example high durability and shock resistance are needed from aggregates used in construction of pavements, roads or bridges.

3.3 Chemical properties

Chemical properties of aggregates mean cleanliness. Aggregates should not contain material or compounds that can weaken the quality of concrete. Humus, organic degradation products, can effect the setting and hardening of concrete and can damage the concrete structure.

Chlorides accelerate the corrosion of steel reinforcement and therefore damage the structure. The chloride content of aggregates used in concrete production has the maximum limit of 0,02 % (Suomen Betoniyhdistys, 2009, 27). Especially when aggregates from underwater sources are used chloride content has to be determined.

Sulphates react inside the concrete forming ettringite that can cause cracks in the structure due its greater volume. Ettringite based expansion is not a problem when concrete is in the phase of plasticity, but expansion in hardened concrete causes cracking. Ettringite is formed also if alcalic-reactive aggregates are used. (Suomen Betoniyhdistys, 2009, 28-30.)

Like water used in concrete production, aggregates should contain neither sugars, oils nor fats, and when white concrete is produced, the amount of ferric compounds is also to be limited. Also the radioactivity of the aggregates excavated from specific parts of Finland needs to be determined. (Suomen Betoniyhdistys, 2009, 29-31.)

4 WATER-CEMENT RATIO

4.1 Water in concrete

Concrete can be seen as a complex material: it is heterogeneous, containing aggregates and mortar, and at the same time it is a triphasic material with solid, liquid and gas elements inside it. This microstructural complexity is the origin of specific properties of concrete, such as creep, shrinkage and cracking.

The water in concrete is present in three forms: chemically bound water, physically bound water in gel pores and strongly ionized free water inside capillary pores. Even though it is commonly thought, concrete does not need to dry out in order to harden. Actually when concrete dries it also stops getting stronger, since concrete, and more specifically the cement in it, needs water for hydration and therefore for hardening. The water amount that cement requires for complete hydration is approximately 25 % of its weight (Verbeck, G. 1956, 138).

In freshly mixed cement paste the space filled with water represents the space available for hydration products. As hydration proceeds the space originally filled with water reduces as the C-S-H gel is produced. C-S-H gel has larger volume than unhydrated cement, and eventually the water space that is not filled with hydration products forms a capillary system inside the paste. The volume of capillary pores varies approximately between 0 % and 40 % in well-cured pastes with a water-cement ratio in the normal range. (Verbeck, G. 1956, 138.) Further hydration reduces the size of capillary network and pores, and since calcium hydroxide ($Ca(OH)_2$) is the byproduct of cement hydration, water inside the capillary pores becomes highly alcalic, creating a pH atmosphere of over 13 inside cement paste and concrete (Finnsementti, 1997, 38). Clean water inside capillary pores freezes below 0 ºC, but usual water in capillary pores is so rich in diluted salts that freezing point has decreased a few degrees, being approximately -3 to -4 ºC.

Also C-S-H gel itself is porous, containing exceedingly small pores with the size of only 0,1-5 nm. In a dense, well hydrated paste, gel pores constitute approximately 20 % of the bulk volume of the hydration product. Also these pores are water-filled, but the water inside these gel pores has notably different characteristics than free water has, such as vapor pressure and mobility. Gel pores retain notable amount of water even at low humidity levels due to physical adsorption forces, meaning that water inside gel pores does not freeze in normal conditions. (Verbeck, G. 1956, 137.)

Illustration 4 represents the ratios of cement which has not reacted, hydration products, capillary pores and gel pores as hydration proceeds. Since gel pores are produced when the cement hydration process takes place, the volume of gel pores increases during the hydration process at the same time when the volume of capillary pores decreases.

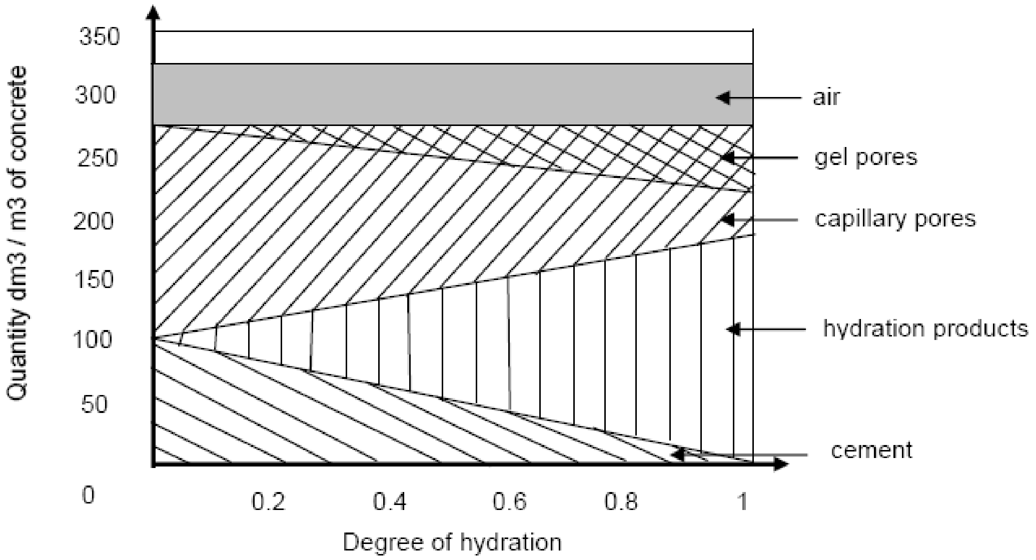


Illustration 4. Combination of unhydrated cement, hydration products, capillary pores, gel pores and air depends on the hydration rate.

4.2 Water in aggregates

Aggregates contain two types of water: absorbed water and free, effective water. In the illustration 5 four different states are represented: oven dry, air dry, saturated but surface dry (SSD), and wet. Finnish standardization system recognizes only two of these conditions, oven dry and SSD (SFS-EN 1097-6, 1999, 3). The primary difference between these two states is the absorption, meaning the pore-filling combination of water and entrapped air.

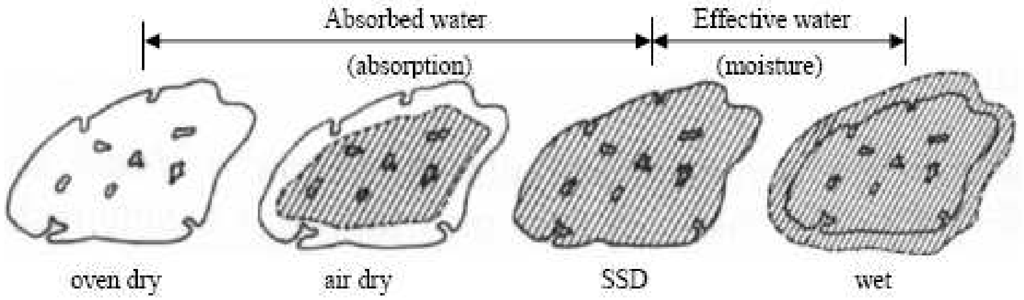


Illustration 5. The primary difference between oven dry and saturated but surface dry (SSD) aggregates is the absorbed water (Suomen Betoniyhdistys, 2005a, 38).

Oven dry aggregates are dried in ventilated oven at 110 °C to constant weight. After this drying period all the water, including absorbed water, is evaporated, aggregate particle pores are empty and particle surfaces are dry. Air dried aggregates are dry from the surface, and also larger pores close to surface are dried. However, pores deeper inside particles remain filled with water. Saturated but surface dried aggregates (SSD) are dry only from the surface, all pores still filled with absorption water and entrapped air. Oven dry, air dry and SSD aggregates are light in color. Wet aggregates have a thin layer of water on aggregate particle surfaces, giving darker color to them. Finer aggregate sizes do not flow when poured, but they cling and form clods under pressure. Dry aggregates do not adhese to each other even under pressure. (Landgren, 1994, 423-424.)

Absorption means the process when small pores inside the aggregate particle imbibe water. The term is used also to describe the mass percentage of water that dry aggregate absorbs during 24-hour period of soaking in water. The weight of the water absorbed is the difference between oven dry and SSD aggregates after soaking period, and the weight of water increases with time of immersion. During the initial phase of absorption the inrush of water forces the air bubbles out from larger pore channels compressing it. This phase occurs quickly making initial absorption rates high. After this initial phase the space that air entrapped deep inside aggregate particle takes can be replaced only by dissolving the air in pore water and transporting it out by slow diffusion. Total absorption consists of this fast initial absorption phase and latter, slow but measurable absorption. (Landgren, R., 1994, 424.) Absorbed water does not participate in cement hydration or affect workability or other properties of concrete. Therefore, if dry aggregates are used in concrete production, the amount of water needed is to be increased by the amount of water that would be absorbed. Absorbed water is

$$m_{aggr,w,abs} = m_{aggr,dry} \times k_A \qquad (1)$$

where $m_{aggr,w,abs}$ is the mass of water absorbed by aggregates [g], $m_{aggr,dry}$ is the mass of oven dry aggregates [g] and k_A is the absorption coefficient, determined to each aggregate type specifically.

The water on aggregate particle surfaces is free water. It is called also effective water, since it can participate in cement hydration reactions and effect fresh concrete properties, such as workability. The total amount of water [g] is

$$m_{aggr,w,total} = m_{aggr,wet} - m_{aggr,dry} \qquad (2)$$

and total moisture percent of aggregates is

$$W_{aggr,total} = \frac{m_{aggr,wet} - m_{aggr,dry}}{m_{aggr,dry}} \times 100\ \% \tag{3}$$

where $m_{aggr,wet}$ is the mass of wet aggregates [g]

If only the amount of effective water [g] needs to be calculated, the following formula can be used

$$m_{aggr,w,eff} = m_{aggr,wet} - (m_{aggr,dry} + m_{aggr,w,abs}) \tag{4}$$

Effective moisture percent [%] is

$$W_{aggr,eff} = \frac{m_{aggr,w,eff}}{m_{aggr,dry} + m_{aggr,w,abs}} \times 100\ \% \tag{5}$$

4.3 Effects of the water-cement ratio

Water-cement ratio is a very important factor in concrete production, and it has crucial effects to both, fresh and hardened concrete properties. Consistency of concrete, meaning its stiffness or fluidity, is usually controlled with water. Different concrete mixes need different amounts of water to reach certain consistency, depending not only on water amount but also on the aggregates (e.g. grain size distribution, the amount of crushed aggregates, absorption coefficients), binders (the amount and type of binders) and admixtures used. The concrete workability is very important during castings; it indicates how easily concrete can be placed in the formwork and compacted. For this concrete needs to be homogeneous and it should not segregate. These properties have connection points to consistency, and can also be controlled with the use of water and admixtures. The most of important factors of good quality concrete construction depend on fresh concrete workability: besides compactness, examples are also fresh concrete cohesion, coating and adherence of concrete to reinforcement steels, hardened concrete strength, resistance, sealing and smooth and desired concrete structure surface. In the following paragraphs some effects of water-cement ratio are introduced.

Segregation and bleeding of fresh concrete are the cases where mixing water moves upward due to settlement of heavier aggregate and cement particles. In concrete structures sedimentation does not occur completely because the chemical reactions between cement and water initiate and the paste starts to stiffen. Nevertheless, in the bottom portion of the concrete lift there can be a loss of water at the same time when on the top part aggregates and the paste are separated leading to an un-homogeneous structure. Water segregation in some amount benefits the successful finishing of some, especially large and horizontal, surfaces, because during setting phase water is also evaporated from the surface. The water evaporation can lead to unsightly plastic shrinkage cracking, if curing is not carried out correctly and there is insufficient amount of water for both, water evaporation and cement hydration. High water-cement ratio and over-wet concrete should not be seen as a solution; segregated water-cement paste on the top surface lead to weakened surface with poorer hardening properties. Obviously, segregation does not affect only fresh concrete properties; it has an effect also to hardened concrete permeability. (Kosmatka, 1994, 88.)

Usually the most important property of concrete is its compressive strength. The strength is directly related to the water-cement ratio, meaning that as the water-cement ratio increases the compressive strength decreases. Water-cement ratio is directly linked to the spacing between cement particles in the cement paste. The smaller the spacing is, the faster cement hydrates fill the gaps between particles, and the links between hydrated particles grow stronger therefore forming a stronger concrete. (Bentz & Aïtcin, 2008, 51.) This is represented in the illustration 6.

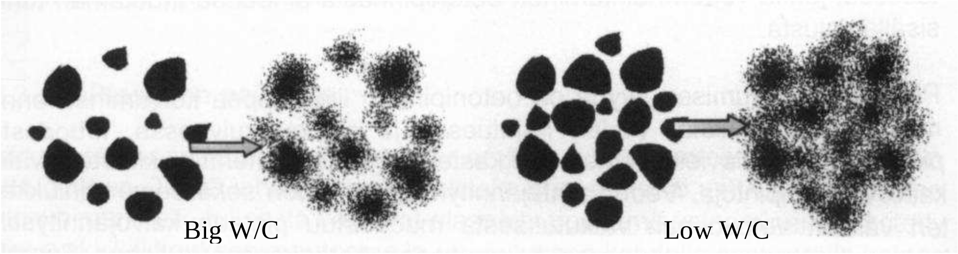


Illustration 6. Lower water-cement ratio means stronger concrete (Suomen Betoniyhdistys, 2005a, 74).

On the other hand, as discussed in chapter 1.2, during the cement hydration heat is evolved and the concrete structure temperature increases. During normal-scale concrete construction heat evolved does not cause a great problem since heat is dissipated into the soil or air. Resulting temperature changes helps to keep a more favorable curing condition and prevents the structure from freezing. In large structures, such as dams and thick foundations or walls, the heat generated inside the structure cannot be readily released and the concrete mass may attain high temperatures. High temperatures causes expansion inside the concrete structure while it is hardening. If the temperature difference within the structure gets too great, for example when the structure undergoes rapid or nonuniform cooling, stresses due to thermal contraction in conjunction with structural restraint can result in cracking of the structure. (Suomen Betoniyhdistys, 2005a, 95.)

Concrete permeability depends significantly on the paste permeability. When intact concrete is exposed to hydrostatic pressure the permeating water needs to pass through the paste and if the paste is of low permeability the concrete will show similar characteristics (Verbeck, 1956, 137). The effect of water-cement ratio does not restrain to water permeability. With a better quality paste concrete can resist chemical attacks, such as acids and salts, better than concrete with high-water ratio and high porosity. For concretes with low porosity, the gas permeability is always greater than the water permeability. (Struble & Hawkins, 1994, 460.)

When hardened concrete elements dry, the C-S-H gel moisture content is decreased, and the gel volume is contracted leading to drying shrinkage of the element. When the concrete structure is returned to higher humidity, moisture is absorbed and the cement gel volume increases again. The single most important factor affecting shrinkage is the amount of water per unit volume of concrete. Meaning, that concrete with a higher consistency shrinks more, because the consistency is increased mainly through a higher water-cement ratio, greater paste quantity or the combination of those two. In practice, the drying shrinkage of concrete is usually between 0,4-0,8 ‰. (Suomen Betoniyhdistys, 2005a, 90.)

While the deformation caused by shrinkage is reversible, the one caused by creep is not. Creep develops during the time when concrete structure sustains a load. The pressure inside concrete forces water out from C-S-H gel and gel is compacted, possibly by rearrangements of chemical bonds. This deformation usually occurs in the direction the force is being applied, and it can be with the same magnitude over time as drying shrinkage. Concrete that exhibits high shrinkage also shows high creep, and these two phenomena are closely related though the connection is not fully understood. Creep continues for a very long time, but the rate of creep decreases continuously. The creep in hardening concrete is significantly greater than the creep in fully hardened concrete. (Suomen Betoniyhdistys, 2005a, 88-89.)

4.4 Calculation of water-cement ratio

As mentioned in chapter 3.1, theoretically the amount of chemically combined water needed for total cement hydration is approximately 25 % of its mass. In addition also gel pores are formed, using water with approximately 20 % of the cement mass. Theoretically the amount of water needed for total hydration is 40 % by mass of cement. This can be expressed also with a water-cement ratio of w/c 0,40. The water that does not participate in cement hydration fills capillary pores, and when water-cement ratio is greater than 0,45 there is always excess water present in the concrete. In reality, capillary pores are formed also when water-cement ratio is below 0,40 because cement hydration never occurs completely. If water-cement ratio is approximately 0,70 capillary pores form a capillary network inside concrete. (Suomen Betoniyhdistys, 2005a, 77.)

Water-cement ratio can be described simply as the mass of effective water ($m_{w,eff}$) divided by the mass of cement (m_{cem}).

$$W/C = \frac{m_{w,eff}}{m_{cem}} \tag{6}$$

Water in concrete can be considered coming from three different sources: aggregates, admixtures and mixing water. Since water-cement ratio is fixed during concrete mix design, the amounts of water from these different sources need to be calculated during batching, so that the requirements for water-cement ratio are fulfilled.

As stated in Eq.(4) the effective moisture content of aggregates is

$$m_{aggr,w,eff} = m_{aggr,wet} - (m_{aggr,dry} + m_{aggr,w,abs})$$

In concrete production, this moisture content needs to be determined for each aggregate fraction separately, and when the proportions of each aggregate sizes are known per unit volume of concrete, the total amount of effective water from certain aggregate size is calculated as follows

$$m_{aggr,w,eff} = \frac{W_{aggr,eff}}{100} \times m_{aggr} \tag{7}$$

where m_{aggr} is the mass of aggregate.

The water from admixtures ($m_{w,adm}$) can be calculated with the information from admixture data sheet, where the mass percentage of dry content (dC) is mentioned. The water from admixture is

$$m_{adm,w} = m_{adm} \times \frac{100 - dC}{100} = m_{adm} \times \left(1 - \frac{dC}{100}\right) \quad (8)$$

where m_{adm} is the mass of admixture used in batching.

The total amount of water is then

$$\begin{aligned} m_{w,total} &= m_{aggr,w,eff} + m_{w,adm} + m_{w,mixing} \quad (9) \\ &= (m_{aggr,w,eff,i} + m_{aggr,w,eff,j} + \ldots) + (m_{w,adm,a} + m_{w,adm,b} + \ldots) + m_{w,mixing} \end{aligned}$$

Where i, j,.. refers to different aggregate sizes and a, b, … refers to different admixtures.

As mentioned in the chapter 1.2, when additives are used the cement amount is calculated with activity factors (k_A). The total cement amount CEM_{Iequ} is

$$CEM_{Iequ} = k_{A,cem} \times m_{cem} + k_{A,add,1} \times m_{add,1} + k_{A,add,2} \times m_{add,2} + \ldots \quad (10)$$

The total water-cement ratio therefore is

$$\begin{aligned} W/C &= \frac{m_{w,total}}{CEM_{Iequ}} \quad (11) \\ &= \frac{m_{aggr,w,eff} + m_{w,adm} + m_{w,mixing}}{k_{A,cem} \times m_{cem} + k_{A,add,1} \times m_{add,1} + k_{A,add,2} \times m_{add,2} + \ldots} \end{aligned}$$

4.5 Determining the actual water-cement ratio

In Finland the requirements for concrete production are defined in Betoninormit BY50, which is based on the European standard EN 206-1. Requirements for water-cement ratio are based on concrete exposure classes, and it is the concrete producers responsibility to follow these regulations and fulfill the requirements set in them. This is not always an easy task to do, especially when special concrete, such as self-compacting concrete or concrete with high consistency, needs to be produced. In cases like these, a difference in water amount as small as 5 kg/m^3 can cause tremendous effects to workability of fresh concrete and later durability of hardened concrete.

When water from admixtures can be calculated precisely, and also the quantity of mixing water can be accurately controlled during batching, the main variation in water amount comes from the aggregates. Aggregate moisture varies throughout a stockpile, with wetter aggregates located near the bottom of the pile, even in constant conditions. Furthermore, transportation, storing conditions and weather have an effect to the moisture of the aggregates. It is required that aggregate moisture is determined at least once a day, but when concrete highly sensitive to variations in the water amount is produced, measurement once a day is not sufficient. In this situation aggregate moistures need to be tested more frequently, especially when changes in consistency occur, or weather conditions change the moisture contents in storage. During concrete production, the testing of aggregate moistures needs to be fast and the results accurate.

4.5.1 Gravimetric methods for determining the aggregate moisture content

Aggregate moisture can be determined using gravimetric or volumetric methods. According to the standardized method described in SFS-EN 1097-5 (1999, 3) the aggregate moisture is determined using a ventilated oven. The obvious disadvantage of this method is the time it requires, approximately 20 hours. A faster method is that where aggregates are dried in a drying pan with gas burners. The test has duration of about 1 hour, results tend to be over-estimated and there is an occupational safety aspect, working with open flames and gas. Both of these methods are gravimetric and they are introduced with more care in the chapter 5.1.1.

4.5.2 Volumetric methods for determining the aggregate moisture content

Batching systems can be equipped with volumetric moisture measurement, probes that read the aggregates moisture content. These systems are typically tied directly with the batching computer and automatically adjust the batch weights to correct proportions and the water-cement ratio. Various systems exist today, based on electromagnetic techniques, radio activity and wave lengths in infra-red or microwave frequency areas.

Electromagnetic techniques include probes measuring capacitance or resistance of the material. Capacitive probes measure capacitance between its two electrodes to determine the dielectric permittivity of its ambient material. This dielectric permittivity is directly proportional with moisture content. However, the measurement is influenced by the aggregate type and temperature, and therefore careful calibration is needed and its stability over time is not good. With resistive probes the resistivity of the material between electrodes is measured. The difficulty with resistive sensors is that the absolute material resistivity depends on ion concentration as well as on moisture content; therefore careful calibration for each aggregate type is required. (Bouygues Construction, 2005, 10.)

By using neutron scattering fast neutrons emitted from a radioactive source are slowed down by hydrogen atoms. Since most of the hydrogen atoms are components of water molecules, the proportion of slowed down neutrons is related to aggregate moisture content. Equipment for this method is relatively costly, and the radioactive source required incurs a radiation hazard. (Bouygues Construction, 2005, 10.)

Systems based on infrared or microwave principals measure the electromagnetic energy that has been either reflected or emitted at distinct wavelengths by the aggregates. The energy absorption depends on the moisture content, but intensity of radiation with moisture can vary depending on dielectric properties and temperature. The near-infrared method measures the molecular absorption only from the surface layer and therefore they are not very applicable if moisture distribution is not homogeneous. (Bouygues Construction, 2005, 10.)

All the above described measuring systems are based on volumetric moisture measurement depending on the density of the material. Aggregate density can be taken as relatively constant factor, but several factors affecting the calculation of aggregate quantity, such as dosing speed and possible dosing errors, are not. For example, with capacitive and resistive probes, the installation and correct directioning of the probe are essential. Usually volumetric probes are installed in aggregate bin wall, and in order to achieve good results the sensing head needs to be in a steady aggregate flow, and not surrounded by compacted or slowly moving aggregates. The correct directioning of the probe in a cross-sectional view of an aggregate bin is represented in the illustration 7.

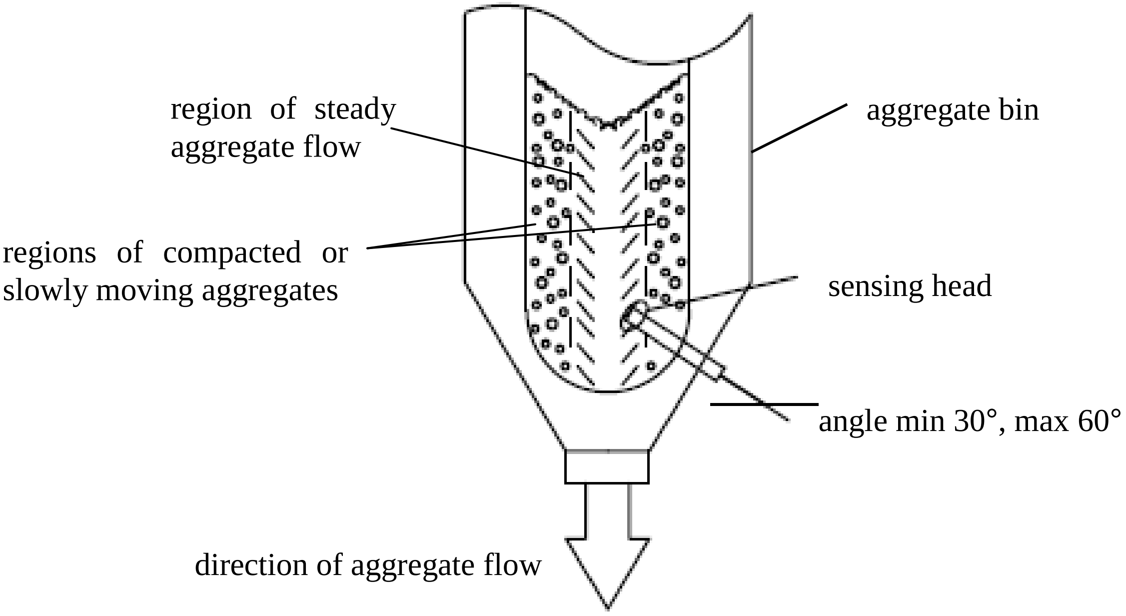


Illustration 7. Proper probe directioning is essential when volumetric moisture measuring systems are used.

As mentioned, installation, maintenance and calibration of these probes can be difficult. Method accuracies have been determined in theory to be between ± 0,5 % and ± 1,0 %, but in practice their reliability and ease of use is not sufficient for concrete production and therefore the use of these methods is rare. (Bouygues Construction, 2005, 10.)

5 OBJECTIVES OF THE RESEARCH

Based on the information about the current situation described in the chapter 4.5 a new method for aggregate moisture measurement needs to be developed. To be suitable for every-day-use in a concrete batching plant the method needs to be simple, easy to use and produce accurate results quickly.

The target of this thesis is to validate the use of microwave ovens in determining the moisture content of aggregates by comparing the microwave oven method to other gravimetric methods (the ventilated oven method and the gas burner method). If the results are found to be reliable, a test protocol will be developed in order to produce reliable results quickly for the use in concrete production.

Parameters to be optimized are the mass of wet sample, total duration of the test, power setting of the microwave oven and the sequence of heating intervals during the testing. The temperature of the sample will also be followed during the test, because it is correlated to the mass of evaporated water, but that is not within main priorities of the research program.

6 REVIEW OF THE RESEARCH

Four standard aggregate sizes were studied: filler 0/1 mm, sand 0/6 mm, crushed aggregates 4/8 mm and 8/16 mm. All measured values were always compared to a reference result values obtained by using ventilated oven drying method as defined in the standard SFS-EN 1097-5.

The test program is divided in 4 main stages, according to the table 4.

Table 4. The test program.

Stage	Description
1	Validation of microwave oven method: general comparison of aggregate moisture results obtained by other gravimetric methods, the ventilated oven method and the gas burner method.
2	Determination of optimized power and heating time: microwave oven power setting [W], total duration of testing [min] and heating intervals [min].
3	Determination of optimum wet sample mass [g].
4	Final procedure simplification.

All samples were collected from covered storage buildings or dosing belt of batching plant silos, all samples were homogeneous, and each sample was divided into fractions and each fraction was tested according to the test program. Tests from the same sample were carried out simultaneously as far as it was possible, and if the sample had to be stored before testing, it was stored accordingly with exact identification in sealed, air-tight container to prevent moisture loss through evaporization. Before testing the sample was remixed to avoid uneven distribution of the moisture.

6.1 Validation of the microwave oven method

In 2008 as a matter of concrete production, it was decided to compare the method used at that time (gas burner method) to the microwave oven method in order to improve quality control. In this stage (validation of the microwave oven method) the test results obtained between June 2008 and August 2008 were used. Aggregate sizes used during this time were 0/1 mm, 0/8 mm and 8/16 mm. Moisture of aggregates was tested simultaneously using three methods: the ventilated oven method, the gas burner method and the microwave oven method.

6.1.1 The reference method: drying by the ventilated oven

According to the standard SFS-EN 1097-5 (1999, 4) minimum mass of wet sample is obtained as follows:

- when maximum aggregate size D ≥ 1,0 mm, the minimum mass of wet sample [kg] is 0,2 D
- when maximum aggregate size D < 1,0 mm, the minimum mass of wet sample is 0,2 kg

Minimum mass for each aggregate size for this test was fixed according to the table 5.

Table 5. Minimum wet sample mass for reference method.

Aggregate size [mm]	Minimum mass [g]
0/1	1000
0/6 (parts 2, 3 and 4)	1200
0/8 (part 1)	1600
4/8 (parts 2, 3 and 4)	1600
8/16	3200

The reference test procedure was as follows: Wet sample is placed as an even layer to the weighed heat-proof pan, and the total mass of pan and sample is recorded. The pan is then placed in the ventilated oven, in (110 ± 5) °C and dried until constant weight is reached. For determination of constant weight the pan is covered with a plastic film to avoid the moisture absorption from the atmosphere and allowed to cool down to room temperature and weighed. After this the pan is put back to oven for at least an hour, and cooling and weighing is repeated. This cycle is continued until the last weight measurement differs less than 0,1 % from the previous weighing. (SFS-EN 1097-5, 1999, 4.) Protective gloves have to be worn when hot pans are handled. The first drying sequence was fixed to last for minimum 20 hours.

The moisture content is calculated as stated in Eq. (3)

$$W_{aggr,total} = \frac{m_{aggr,wet} - m_{aggr,dry}}{m_{aggr,dry}} \times 100\ \%$$

The result is rounded to nearest 0,1 %. For determining the difference between hot and cooled down samples, in the stages 2, 3 and 4 all reference samples were also weighed directly after being taken out from the oven.

Equipment used for the reference method are represented in the table 6.

Table 6. Equipment used for the reference method.

Reference method, equipment used
ventilated oven at 110 ± 5 °C, Memmert UFE800
weighing scale: KERN 572-49, maximum weight 16,1 kg, accuracy 0,1 g (VTT Expert Services Oy, 2009a).
drying pan made of steel
plastic buckets with lids as airtight and sealable containers, to prevent the moisture loss
tape and permanent marker
trowel
protective gloves

Photos of equipment that was used for testing are represented in the appendix 1.

6.1.2 The gas burner method

Minimum mass of wet aggregate samples was to be the same than as that of the reference method: 1000 g for 0/1 mm, 1600 g for 0/8 mm and 3200 g for 8/16 mm. The test procedure was as follows: Wet sample is placed on pre-weighed, heat- and flame-proof pan, and the total mass of pan and sample is recorded. The pan is placed on gas burner and stirred with trowel or spatula until no steam or aggregate clots cannot be seen. After this the total mass is recorded again, the pan is placed back on the burner and the drying and weight recording is repeated until constant mass (differing < 0,1 %) is reached. Protective goggles and gloves have to be worn during the drying with gas burners. Equipment used for the reference method are represented in the table 7. Differing from the reference method, the sample is not allowed to cool down, because the aim is to determine the moisture content as fast as possible.

Table 7. Equipment used for the gas burner method.

Gas burner method, equipment used
gas burner, Lincar 1312BQ4242, with safe connecting valves and pipes to gas bottle
propane
powered ventilator
weighing scale: KERN DS360,5, maximum weight 36,0 kg, accuracy 0,5 g (VTT Expert Services Oy, 2009b).
drying pan made of steel
plastic buckets with lids as airtight and sealable containers, to prevent the moisture loss
tape and permanent marker
trowel
lighter
protective gloves and goggles

Photos of equipment that was used for testing are represented in the appendix 1.

6.1.3 The microwave oven method

While using the microwave oven method, two different standards used in geology were studied during the test period: NF P94-049-1 and ASTM D 4643-08. These two standards for soils have almost the same test procedure; the ASTM standard requires a lower wet sample mass and allows lower microwave oven power. The main differences between these two standards are shown in the table 8.

Table 8. Main differences between standards NF P94-049-1 and ASTM D 4643-08 (NF P 94-049-1, 1996, 5-6 & ASTM D-4643-08, 2008, 3).

	Microwave oven power	**Mixing when weighed**	**Mass of wet sample**	
NF P94-049-1	Minimum 1500 W	no	maximum aggregate diameter [mm]	mass [g]
			1	200
			5	300
			6.3	400
			8	500
			16	2000
ASTM D 4643 - 08	Suggests the highest power setting	yes	sieve retaining ≤ 10 % of sample [mm]	mass [g]
			2	100-200
			4.75	300-500
			19	500-1000

For this test minimum wet sample mass for each aggregate size were determined according to the table 9.

Table 9. Minimum mass of wet sample for microwave oven method.

Aggregate size [mm]	Minimum mass [g], NF P94-049-1	Minimum mass [g], ASTM D 4643-08
0/1	250	100
0/8	500	400
8/16	2000	500

Procedure for the microwave oven method was as follows:

Wet sample is placed to pre-weighed, microwave oven-durable dish, and total mass of pan and sample is recorded. The dish is heated in microwave oven (power setting 1600 W) for at least 3 minutes, dry weight is recorded and the dish is re-heated in the microwave oven for 3 minutes and weighed. When the ASTM method was tested samples were stirred with spatula before weighing. After this a cycle of 1 minute-heating and weighing is repeated until constant mass is reached. Protective gloves have to be worn when hot dishes are handled. Equipment used for the microwave method are represented in the table 10.

Table 10. Equipment used for the microwave oven method.

Microwave oven method, equipment used
microwave oven: Panasonic Pro II NE-3240, maximum power 3200 W
weighing scale: KERN 572-49, maximum weight 16,1 kg, accuracy 0,1 g (VTT Expert Services Oy, 2009a).
microwave oven durable dishes (Pyrex)
thermal-shock resisting coaster, produced from e.g. cork
plastic buckets with lids as airtight and sealable containers, to prevent the moisture loss
tape and permanent marker
hand shovel and spatula
protective gloves

Photos of equipment that was used for testing are represented in the appendix 1.

6.2 Determination of optimized power and heating time

Recommendations and guidelines for the aggregate moisture determination were taken from the two soil standards tested in the previous stage (NF P94-049-1 and ASTM D 4643-08). In this stage the general test procedure was examined with more care, using the information gained from the previous stage. The target was to determine a procedure that would act as a basis for further testing, focusing on microwave oven power and on total test duration and their impact to sample temperature during drying process.

Altogether 3 test series, each with 10 samples, were carried out for each aggregate size: 1 series with the original procedure determined in the stage 1, 1 series with full power (3200 W) and 1 series with half power (1600 W) and a longer first heating period, and series 3 with the procedure as described determined in the previous stage. The aim of having different heating intervals was in checking the influence of the number of mixing times to the total test duration. The amounts of minimum wet sample mass were derived from the pervious stage, and those are represented in the table 11.

Table 11. Stage 2: Minimum mass of wet sample.

Aggregate size [mm]	Minimum mass [g]
0/1	100
0/6	300
4/8	400
8/16	500

Microwave oven power was kept at a constant in each series, while the duration of heating intervals varied in order to find the optimal procedure. In series 3 heating intervals were kept constant. When this stage was started, heating intervals were as follows:

Series 2: 0/1 mm & 0/6 mm: 7 min + 5 min + 3 min + 1 min + 1 min + …
4/8 mm & 8/16 mm: 5 min + 5 min + 2 min + 1 min + 1 min + ...
Series 3: 0/1 mm & 0/6 mm: 2 min + 2 min + 2 min + 1 min + 1 min + ...
4/8 mm & 8/16 mm: 2 min + 2 min + 1 min + 1 min + ...

After testing, the optimum set of parameters was determined as it is represented in the table 12.

Table 12. Determined parameters.

Parameter	Series 1		Series 2		Series 3	
Power [W]	1600		1600		3200	
Heating sequence [min]	0/1 mm & 0/6 mm	5+3+2+1 +1+…	0/1 mm & 0/6 mm	10+1+1+…	0/1 mm & 0/6 mm	5+3+2+1+ 1+…
	4/8 mm & 8/16 mm	3+3+2+1 +1+…	4/8 mm & 8/16 mm	7+1+1+…	4/8 mm & 8/16 mm	3+3+2+1+ 1+…
Mixing	yes		yes		yes	

Samples were dried until constant mass was reached, and all samples were weighed directly after being removed from the microwave oven and again after cooling.

6.3 Determination of optimum wet sample mass

In this stage, in order to totally optimize the procedure, the optimum wet sample mass was determined with the most suitable method determined in the previous stage. 3 series, each consisting 10 samples, were prepared for each aggregate size. Each series had different wet sample mass with 100 g increments as represented in the table 13. In this stage parameters used were as in the series 2, stage 2 (table 12).

Table 13. Minimum mass of wet sample.

Aggregate size	Series 1 [g]	Series 2 [g]	Series 3 [g]
0/1 mm	100	200	300
0/6 mm	300	400	500
4/8 mm	200	300	400
8/16 mm	300	400	500

Samples were dried until constant mass was reached, and all samples were weighed directly after being removed from the microwave oven and again after cooling.

6.4 Final simplifying of the procedure

In the last part the aim was to simplify the procedure: it was done by checking the influence of mixing the aggregate sample during the test. 10 samples for each aggregate size were tested. The wet sample was dried in the microwave oven only once and afterwards the dried sample was weighed without stirring. As it will be shown in the chapter 7.4, results obtained for the 4/8 mm sized aggregate using this method were not satisfactory, and the testing of 4/8 mm was continued with two other series. In the second series the wet sample mass was increased, otherwise the procedure was kept the same. In the last series the original wet sample mass was used, but the dry sample was mixed with spatula before weighing. 10 samples were tested also for series 2 and 3. All samples were weighed directly after being removed from the microwave oven and again after cooling. Parameters for this stage are represented in the tables 14 (power, heating and mixing) and 15 (wet sample mass).

Table 14. Microwave oven power, heating intervals and stirring.

Parameter	Stage 4	
Power [W]	1600	
Heating sequence [min]	0/1 mm & 0/6 mm	10+1+1 +…
	4/8 mm & 8/16 mm	7+1+1+ …
Stirring	Series 1&3: no, series 2: yes	

Table 15. Minimum mass of wet sample.

	0/1 mm	0/6 mm	4/8 mm	8/16 mm
Minimum mass [g]: Series 1, no stirring	300	400	200	300
Minimum mass [g]: Series 2: no stirring	-	-	400	-
Minimum mass [g]: Series 3: stirring	-	-	200	-

Temperature measurement

Surface temperature was recorded from microwave oven dried samples every time that samples were weighed. Temperature was followed with infrared thermometer, which stores information over measuring period. Maximum temperature and average temperature were recorded during approximately 10 seconds following the path represented in the illustration 8.

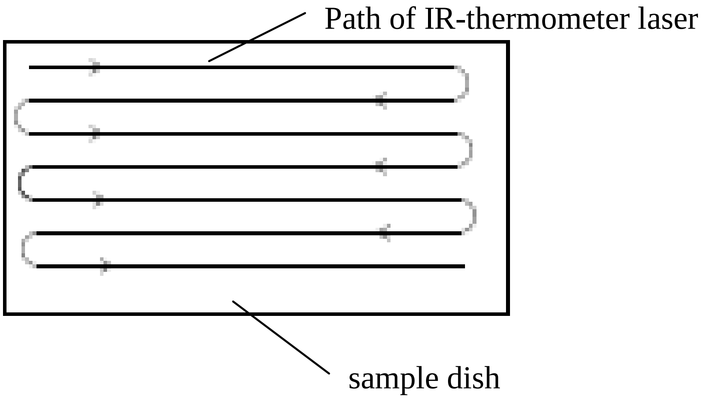


Illustration 8. The whole sample dish area was covered during the temperature measurement.

The equipment used for the temperature measurement is represented in the table 16.

Table 16. Equipment used for the temperature measurement.

Temperature measurement, equipment used
digital thermometer: Fluke 54II, accuracy 0,1 °C (Harrico PTE Oy, 2009).
probe PK29M (Comark Limited, 2009).
infrared thermometer: Fluke 66

Photos of equipment that was used for testing are represented in the appendix 1.

7 RESULTS AND ANALYSIS

In this chapter the obtained results are represented and analyzed.

7.1 Validation of the microwave oven method

As described in the chapter 6.1 results obtained in year 2008 were used to compare microwave oven and gas burner as drying method. Two standards for testing soils were applied for the microwave oven method: NF P94-049-1 and ASTM D 4643-08. They differ mainly in used microwave oven power and wet sample sizes.

Due to the relatively large number of test results for this part they are shown in the appendices 2 and 3, only the statistical analysis of the results is represented in this chapter. Drying results with according to NF P94-049-1 are represented in the appendix 2 and results using the method in ASTM D 4643-08 in the appendix 3. As an overview, the statistical analysis of the test results is represented below, in the table 17.

Table 17. Statistical analysis of measured moistures.

		Min [%]	**Max [%]**	**Average [%]**	*St.dev.*
0/1 mm, ASTM	**Oven**	7.5	9.4	**8.4**	***0.6***
	Burners	8.4	10.6	**9.4**	***0.6***
	Microwave	7.5	9.9	**8.8**	***0.7***
0/1 mm, NFP	**Oven**	7.2	9.2	**8.1**	***0.7***
	Burners	8.3	10.4	**9.4**	***0.7***
	Microwave	7.8	9.6	**8.6**	***0.6***
0/8 mm, ASTM	**Oven**	4.6	6.3	**5.2**	***0.6***
	Burners	5.4	7.2	**6.1**	***0.6***
	Microwave	4.8	6.6	**5.4**	***0.6***
0/8 mm, NFP	**Oven**	3.8	5.9	**4.9**	***0.6***
	Burners	4.4	6.7	**5.8**	***0.5***
	Microwave	4.1	6.4	**5.4**	***0.6***
8/16 mm, ASTM	**Oven**	0.1	1.3	**0.9**	***0.4***
	Burners	0.5	1.8	**1.1**	***0.4***
	Microwave	0.1	1.6	**0.9**	***0.4***
8/16 mm, NFP	**Oven**	0.1	1.1	**0.7**	***0.3***
	Burners	0.6	1.4	**1.0**	***0.2***
	Microwave	0.3	1.3	**0.8**	***0.3***

Measuring the moisture content of aggregates by ventilated oven is the standardized method in Europe. Due to the 20 hour duration of this method it is impractical to use during the concrete production. For this reason the use of gas burners and microwave ovens were tested in addition to standardized method. Differences between tested drying methods compared to the reference method are represented in the appendix 4. It includes two separate tables, where microwave oven methods NF P94-049-1 and ASTM D 4643-08 are represented in their own tables. Statistical analysis of deviations in percentage units is shown below, in the table 18 as conclusion.

Table 18. Statistical analysis of deviations between the test method and the reference method.

		0/1		0/8		8/16	
		Oven Burners	**Oven Microwave**	**Oven Burners**	**Oven Microwave**	**Oven Burners**	**Oven Microwave**
ASTM	**Average**	1.0	0.4	0.9	0.2	0.3	0.1
	St.dev.	*0.3*	*0.2*	*0.2*	*0.2*	*0.2*	*0.1*
NFP	**Average**	1.1	0.4	0.9	0.4	0.3	0.1
	St.dev.	*0.2*	*0.3*	*0.2*	*0.2*	*0.1*	*0.1*

Drying with gas burners is significantly faster than reference method; with gas burners it takes approximately 45 minutes to dry the aggregates. Nevertheless, significant deviations between the results from the reference method and the burners can be seen. The gas burner method kilns the aggregate at high temperature during a relatively long time, causing an overestimation to measured moisture contents. This overestimation can be seen from the table 18: for example with 0/1 mm aggregate size the moisture measured with the gas burner method in ASTM test series was in average 1,0 percentage unit higher than it was measured with the reference method. This case is illustrated in the table 18 by high-lighting it with yellow colour.

The results obtained by the microwave oven method (NF-P 94-049-1) are more representative and also faster: drying takes approximately 15 – 20 minutes**.** Moisture results are still overestimated, as can be seen from the table 18. The results obtained by the microwave oven method (ASTM D 4643-08) are slightly better than the microwave oven method (NF-P 94-049-1). Drying with ASTM D 4643-08 takes approximately 15 minutes being slightly faster than the method in NF-P 94-049-1.

The standard deviations comparing methods of burners and the microwave oven to the reference method are significant, 0,2. This aspect can be explained by the fact that with the reference method samples are allowed to cool down to the room temperature before weighting. Although the reference samples were covered with plastic to limit the absorption of water from the atmosphere, the absorption still occurred and had an impact to the results.

Conclusions from the preliminary tests

From these first results obtained it can be seen that microwave oven is significantly faster and it also provides more accurate results than other methods. Therefore the microwave oven method is more suitable than the gas burner method as a testing method in concrete production. The accuracy of the results gets better when coarseness of aggregates increases: deviations (collected from the table 18) to the reference method with the gas burner and the ASTM microwave oven methods as percentage units are shown in the table 19.

Table 19. Average differences: the reference method compared to the gas burner method and the reference method compared to the ASTM microwave oven method

Aggregate size [mm]	**Oven Burners**	**Oven Microwave (ASTM)**
0/1	1.0	0.4
0/8	0.9	0.2
8/16	0.3	0.1

Standard deviations with both tested methods were the same (0,2), but when overall differences with reference and microwave oven method were smaller, also the maximum difference to reference method remains smaller.

7.2 Determination of optimized power and heating time

Altogether 3 test series for microwave oven, each with 10 samples, were carried out for each aggregate size: series 1 with the procedure determined in the stage (1600 W), series 2 with half power (1600 W) and a longer first heating sequence, and series 3 with full power (3200 W). A sample using the reference method was also tested, and all samples were weighed directly after being removed from the microwave oven and again after cooling. All measured moistures in this stage are represented in the table 20.

Table 20. Measured moistures.

Aggregate size [mm]	**Sample**	**Reference method**		**Series 1 (ASTM D 4643-08)**		**Series 2 (1600W, 1st drying period long)**		**Series 3 (3200W)**	
	number	**Oven, hot [%]**	**Oven, cool [%]**	**Micro-wave, hot [%]**	**Micro-wave, cool [%]**	**Micro-wave, hot [%]**	**Micro-wave, cool [%]**	**Micro-wave, hot [%]**	**Micro-wave, cool [%]**
0/1	**1**	8.3	**8.0**	8.4	7.9	8.5	8.1	8.3	7.8
	2	8.4	**7.9**	8.5	8.0	8.3	7.9	8.3	-
	3	8.5	**8.2**	8.2	7.7	8.4	7.9	8.6	8.0
	4	9.3	**9.0**	9.4	8.9	9.5	8.8	9.4	8.9
	5	9.1	**8.7**	9.5	8.9	9.0	8.5	9.6	9.1
	6	8.6	**8.3**	8.4	8.0	8.3	8.0	8.8	8.2
	7	8.6	**8.2**	8.5	8.1	8.7	7.9	8.6	8.2
	8	9.9	**9.6**	9.9	9.6	9.5	9.2	10.3	9.9
	9	8.7	**8.3**	8.6	8.1	8.5	8.1	8.6	8.2
	10	9.8	**9.5**	10	9.6	9.8	9.2	9.8	9.4
0/6	**1**	5.0	**4.6**	5.0	4.4	5.0	4.4	5.0	4.4
	2	5.0	**4.5**	5.1	4.5	4.5	4.5	5.1	-
	3	4.9	**4.4**	5.0	4.5	5.2	4.6	5.3	4.5
	4	5.6	**5.1**	5.6	5.1	5.7	5.0	6.0	5.3
	5	5.4	**4.9**	5.3	4.8	5.5	4.9	5.6	5
	6	7.6	**7.1**	7.7	7.3	7.7	7.2	7.7	7.1
	7	5.6	**5.1**	5.4	4.9	6.0	5.2	5.9	5.2
	8	5.3	**4.8**	5.4	5.0	5.5	4.8	5.6	4.9
	9	5.3	**4.8**	5.5	4.9	5.6	4.9	5.5	4.8
	10	5.6	**5.1**	5.7	5.2	5.6	5.2	5.9	5.2
4/8	**1**	2.4	**2.2**	1.8	1.7	2.0	1.9	2.0	1.7
	2	2.3	**2.1**	2.0	1.9	2.0	1.9	2.0	-
	3	1.3	**1.2**	1.2	1.1	1.5	1.3	1.5	1.3
	4	2.5	**2.4**	2.5	2.3	2.1	1.9	2.4	2.3
	5	2.4	**2.3**	2.4	2.2	2.4	2.2	2.5	2.3
	6	1.7	**1.6**	1.5	1.4	1.2	1.1	1.7	1.4
	7	1.6	**1.5**	1.6	1.5	1.7	1.4	1.7	1.5
	8	2.7	**2.5**	3.0	2.9	2.7	2.5	3.0	2.8
	9	1.6	**1.4**	1.4	1.3	1.4	1.3	1.6	1.3
	10	2.3	**2.1**	2.5	2.4	2.3	2.1	2.5	2.3
8/16	**1**	0.7	**0.6**	0.7	0.6	0.6	0.6	0.7	0.6
	2	0.7	**0.6**	0.7	0.6	0.7	0.5	0.7	-
	3	0.4	**0.4**	0.5	0.4	0.6	0.5	0.7	0.6
	4	0.7	**0.7**	0.7	0.6	0.8	0.7	0.8	0.7
	5	0.7	**0.6**	0.7	0.7	0.8	0.7	0.9	0.8
	6	1.5	**1.4**	1.4	1.3	1.3	1.2	1.5	1.3
	7	1.6	**1.5**	1.6	1.5	1.7	1.6	2.1	2
	8	0.6	**0.2**	0.3	0.3	0.3	0.2	Testing broken off	
	9	1.8	**1.7**	1.4	1.4	1.5	1.4		
	10	0.4	**0.3**	0.4	0.3	0.4	0.3		

The series 3 with 8/16 mm aggregate had to be broken off due to their extremely high temperatures. These temperatures are shown the appendix 5, and results are analyzed in more details later in this chapter.

The most important parameters in this stage were microwave oven power and drying time, and their impact to the temperature of the samples. These were carefully observed and analyzed, to ensure that the best method could be chosen for following parts of the test program. These measurements are represented in the appendix 5. In the appendix 5 maximum and average sample temperatures, and durations of microwave oven heating and total testing are represented by series. Total time is calculated using the heating time: 2 minutes is added for wet sample weighing and 45 seconds for sample weighing. The total time does not take into account the sampling from the storage area or batching plant silo.

The statistical analysis of results from this stage are concluded in the table 21. Values for minimum, maximum and average moisture differences as percentage units and standard deviations are shown between the reference method and the microwave oven method.

Table 21. Statistical analysis of moisture results.

		Series 1 (ASTM D 4643-08)		**Series 2 (1600W, 1st drying period long)**		**Series 3 (3200W)**	
		reference microwave, hot	**reference microwave, cool**	**reference microwave, hot**	**reference microwave, cool**	**reference microwave, hot**	**reference microwave, cool**
0/1 mm	**Min**	0.0	-0.5	-0.1	-0.4	0.3	-0.2
	Max	0.8	0.2	0.5	0.1	0.9	0.4
	Average	**0.4**	**-0.1**	**0.3**	**-0.2**	**0.5**	**0.0**
	St.dev.	*0.2*	*0.2*	*0.2*	*0.2*	*0.2*	*0.2*
0/6 mm	**Min**	0.3	-0.2	0.0	-0.2	0.4	-0.2
	Max	0.7	0.2	0.9	0.2	0.9	0.2
	Average	**0.5**	**0.0**	**0.6**	**0.0**	**0.7**	**0.1**
	St.dev.	*0.2*	*0.1*	*0.3*	*0.1*	*0.1*	*0.2*
4/8 mm	**Min**	-0.4	-0.5	-0.4	-0.5	-0.2	-0.5
	Max	0.5	0.4	0.3	0.1	0.5	0.3
	Average	**0.1**	**-0.1**	**0.0**	**-0.2**	**0.2**	**0.0**
	St.dev.	*0.2*	*0.2*	*0.2*	*0.2*	*0.3*	*0.3*
8/16 mm	**Min**	-0.3	-0.3	-0.2	-0.3	0.1	-0.1
	Max	0.1	0.1	0.2	0.1	0.6	0.5
	Average	**0.0**	**0.0**	**0.1**	**0.0**	**0.2**	**0.1**
	St.dev.	***0.2***	***0.2***	***0.1***	***0.1***	***0.1***	***0.1***

The method used in the series 3 gives the biggest average deviation compared to the reference method with all aggregate sizes. Otherwise measured moistures do not differ significantly from each other between different methods. The methods used in the series 1 and 2 gave very similar results and no difference were either with standard deviations.

In addition to the measured moistures, the main factors in this stage were the microwave oven power and the drying time, and their impact on sample temperature. Testing of a sample was carried out as long as constant mass was achieved. This gave variations to heating times and total test durations. Statistical analyses of temperature and time recordings from the appendix 5 are represented in the table 22.

Table 22. Statistical analysis of heating times, total test durations and temperature readings.

		Series 1 (ASTM D 4643-08)			
		Heating	**Total**	**Max temp.**	**Avg temp.**
		[min]	[min]	[°C]	[°C]
0/1 mm	**Min**	10.0	13.4	79.7	64.6
	Max	13.0	15.9	137.5	120.8
	Average	**11.1**	**14.5**	**102.3**	**83.7**
0/6 mm	**Min**	10.0	12.9	109.6	86.2
	Max	13.0	16.4	164.2	124.4
	Average	**11.1**	**14.4**	**144.5**	**106.6**
4/8 mm	**Min**	8.0	10.9	119.4	76.8
	Max	11.0	14.8	169.9	116.2
	Average	**9.5**	**12.9**	**142.2**	**100.6**
8/16 mm	**Min**	8.0	10.9	237.9	135.3
	Max	12.0	15.8	407.1	263.4
	Average	**9.6**	**13.1**	**306.1**	**205.8**
		Series 2 (1600W, 1st drying period long)			
0/1 mm	**Min**	11.0	13.9	106.5	84.3
	Max	18.0	21.4	153.3	105.8
	Average	**14.0**	**17.1**	**121.2**	**96.7**
0/6 mm	**Min**	11.0	13.5	159.7	117.0
	Max	20.0	23.4	203.1	148.9
	Average	**14.4**	**17.4**	**177.4**	**129.6**
4/8 mm	**Min**	8.0	10.5	132.8	94.9
	Max	17.0	17.0	267.7	232.3
	Average	**10.7**	**13.6**	**173.3**	**125.9**
8/16 mm	**Min**	8.0	10.5	251.4	168.7
	Max	13.0	15.9	552.2	292.6
	Average	**10.7**	**13.5**	**328.3**	**224.7**
		Series 3 (3200 W)			
0/1 mm	**Min**	6.0	8.9	90.4	70.7
	Max	11.0	13.9	257.9	163.5
	Average	**8.3**	**11.5**	**151.6**	**117.7**
0/6 mm	**Min**	6.0	8.9	162.7	130.4
	Max	11.0	13.9	284.6	182.8
	Average	**8.3**	**11.2**	**225.1**	**159.4**
4/8 mm	**Min**	5.0	7.9	192.7	131.1
	Max	9.0	12.8	305.2	212.7
	Average	**7.4**	**10.8**	**247.9**	**163.0**
8/16 mm	**Min**	5.0	7.9	421.7	273.5
	Max	8.0	11.8	543.0	395.3
	Average	**7.3**	**10.6**	**456.9**	**320.9**

Total test durations by aggregates size (Appendix 5) are represented in the illustrations 8, 9, 10 and 11.

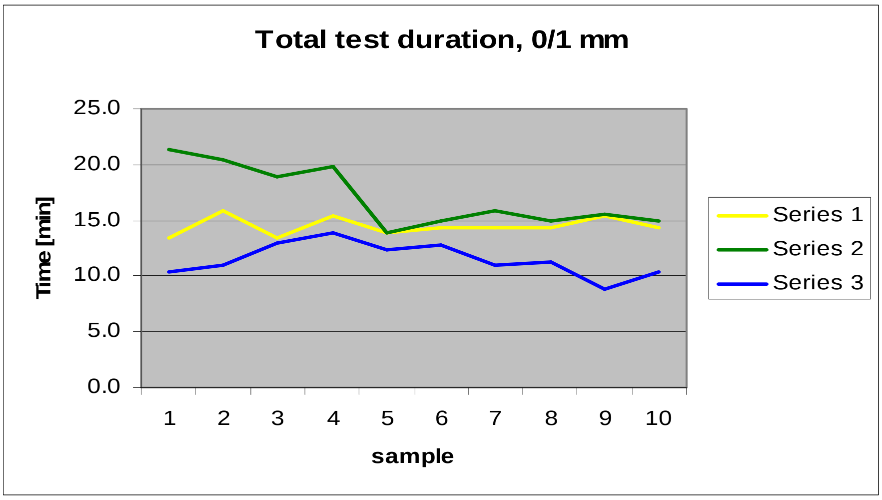


Illustration 8. Total test duration, 0/1 mm.

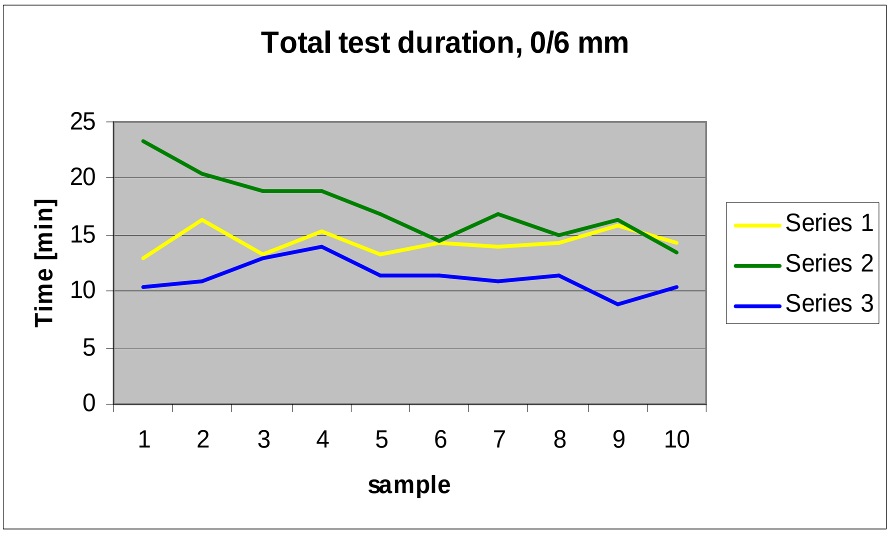


Illustration 9. Total test duration, 0/6 mm.

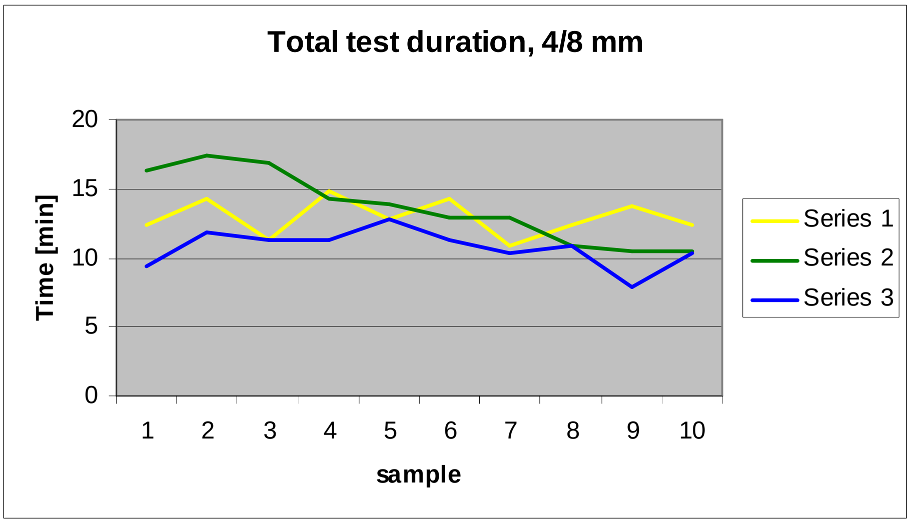


Illustration 10. Total test duration, 4/8 mm.

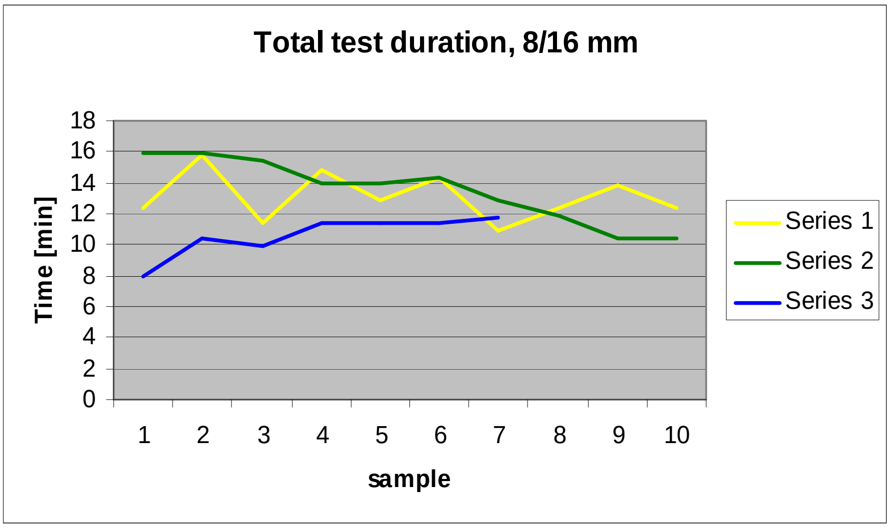


Illustration 11. Total test duration, 8/16 mm.

As it was expected the total test duration was the shortest in the series 3 due to double power in watts used. This had a clear effect on the sample temperatures, as will be shown later. Averages for total test duration in series 2 (table 22) are not completely fair, since as it can be seen from illustrations 8-11, test durations in the first half were several minutes longer than in the latter half. It took a few tests to find out the combination of optimum heating sequences, and more representative values for average durations can be calculated from last 5 samples tested. These values, in addition to average durations from the series 1, are represented in the table 23.

Table 23. Average test durations, modified for series 2.

Aggregate size [mm]	**Average duration [min] Series 1**	**Average duration [min] Series 2**
0/1	14.5	15.2
0/6	14.4	15.2
4/8	12.9	11.5
8/16	13.1	12.0

From the table 23 it can be seen that there is no significant difference in test durations between series 1 and 2. Method in the series 2 seems to be slightly faster with 0/1 mm and 0/6 mm, but slower with 4/8 mm and 8/16 mm.

Temperature

In illustrations 12-17 the temperature information from the appendix 5 is shown in graphic forms. In addition to tested temperatures, safety limits of 350 °C for maximum temperatures and 300 °C for average temperatures were imposed. The purpose of these safety limits will be explained later in this chapter.

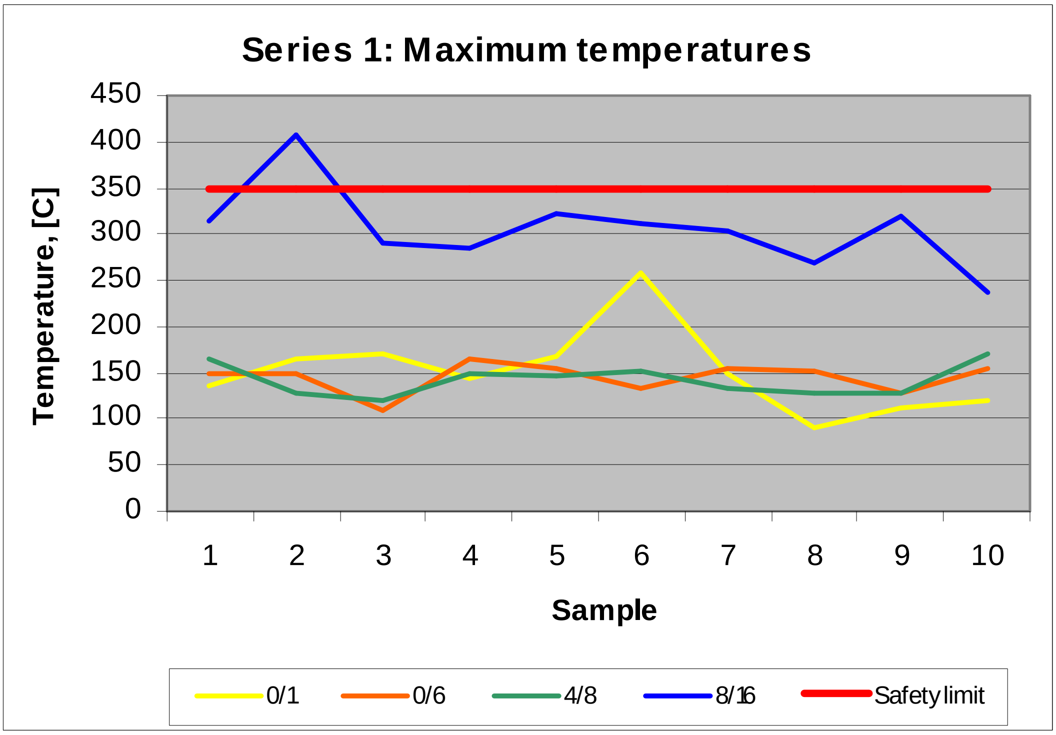


Illustration 12. Series 1: maximum temperatures.

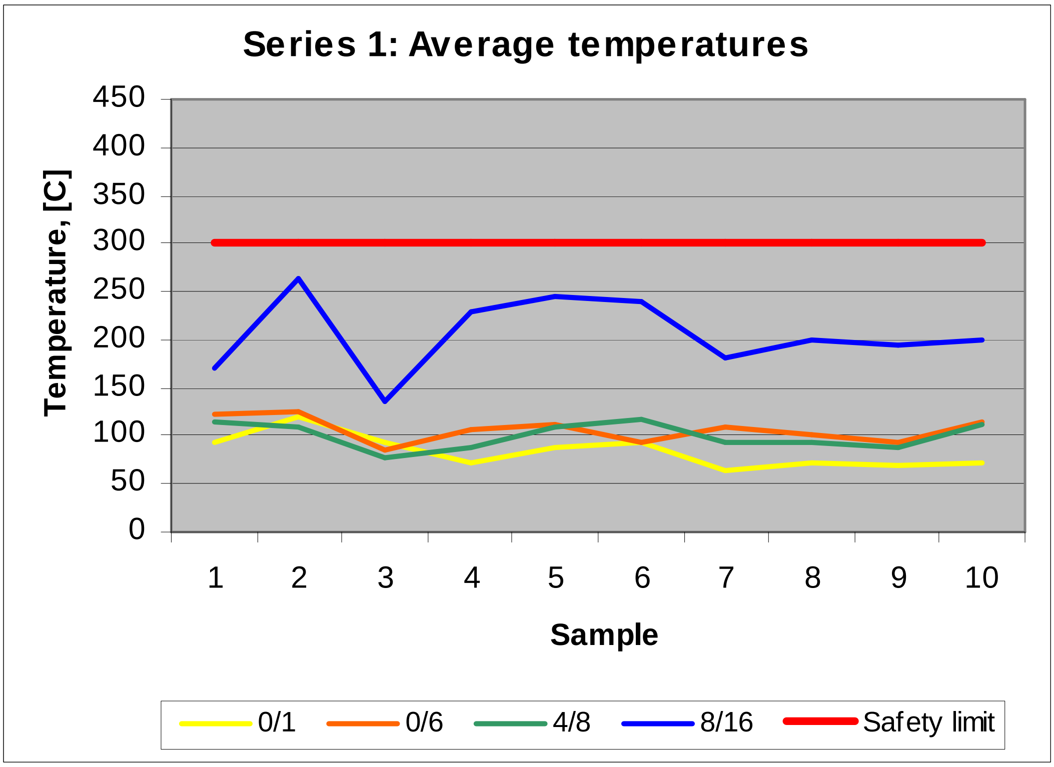


Illustration 13. Series 1: average temperatures.

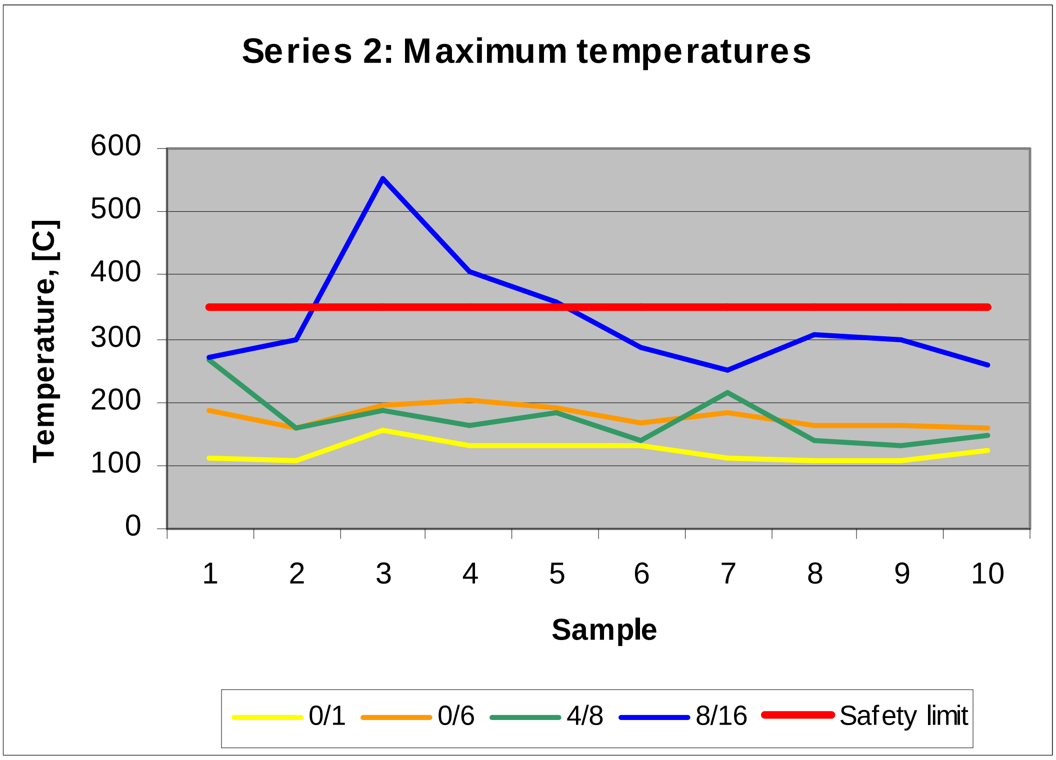


Illustration 14. Series 2: maximum temperatures.

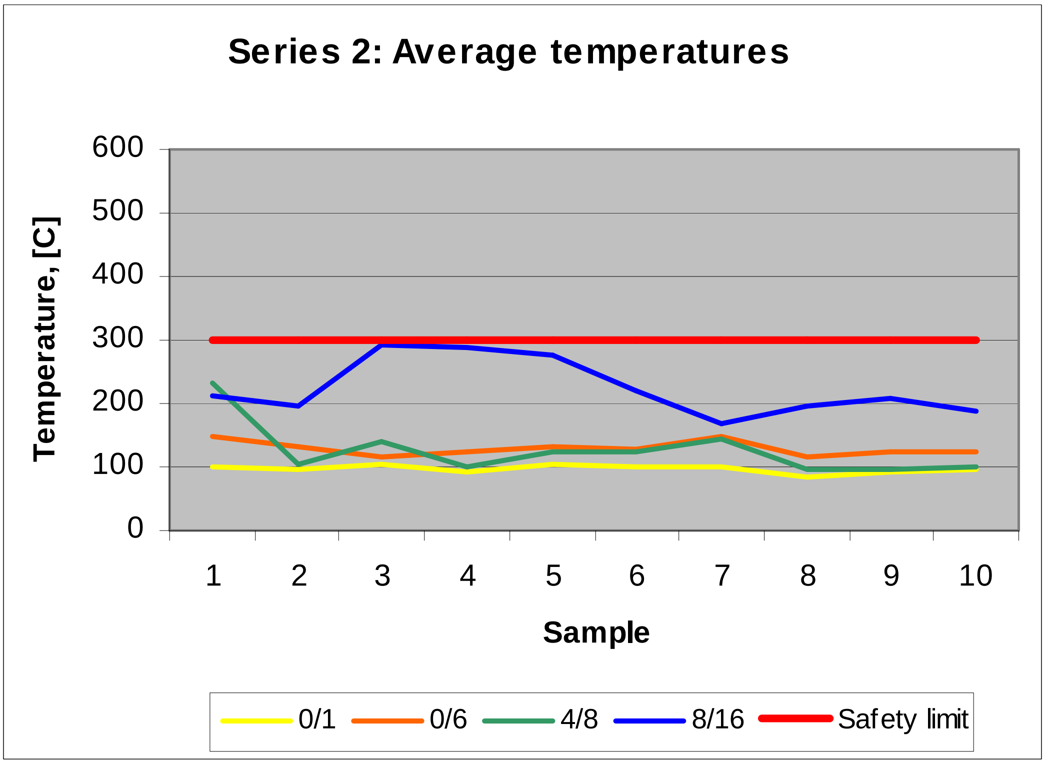


Illustration 15. Series 2: average temperatures.

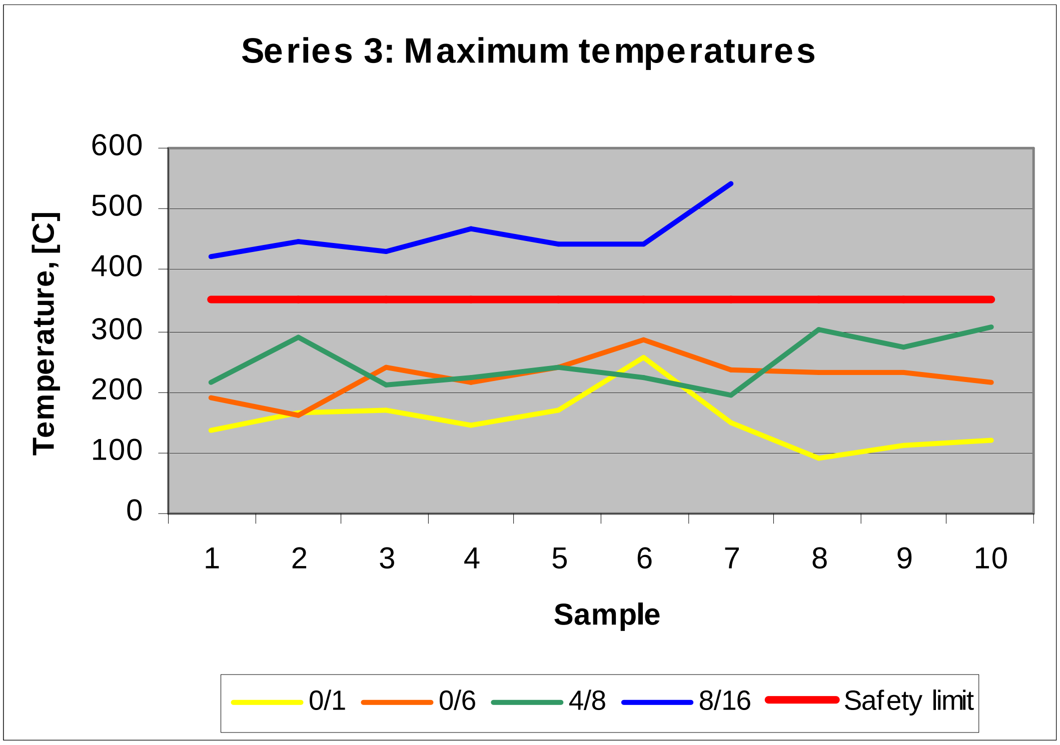


Illustration 16. Series 3: maximum temperatures.

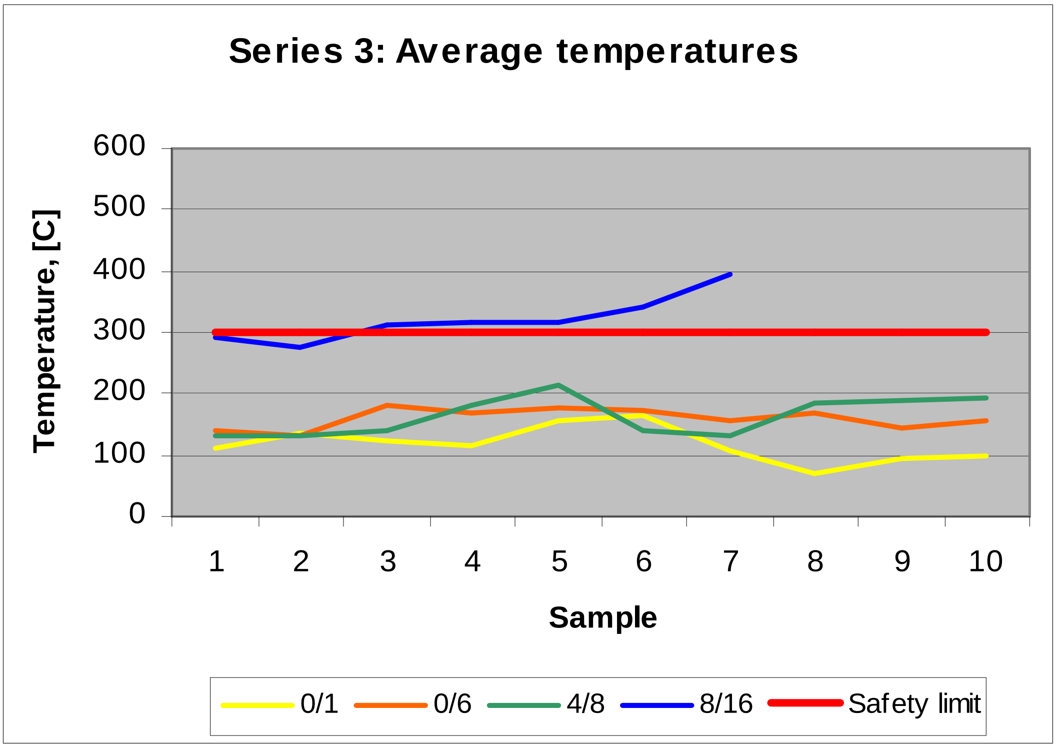


Illustration 17. Series 3: average temperatures.

Glass dishes used for microwave oven drying are exposed frequently to fast heating to very high temperatures and thermal shocks caused by rapid cooling. Due to these reasons glassware sometimes breaks during or after drying. A photo of dish broken by temperature shock is represented below, as the photo 1.

Photo 1. A dish broken after drying test due to thermal shock.

Due to the often breakages the glassware used for tests were inexpensive Pyrex kitchen cookware dishes commonly available in almost every grocery store. Pyrex glass is made of borosilicate glass which is highly resistive to water and different chemicals and has high usage temperatures. According to commercial web site of Duran group, manufacturer of laboratory borosilicate glassware, it is mentioned that maximum operating temperature for DURAN® is 500°C, above 525°C glass starts to soften and above 860°C it turns into liquid state. Cookware glass dishes used do not have the same standard of purity and the quality is not as high as in laboratory glassware; therefore safety limits were set significantly lower. High temperatures tested in this part already left some marks to the glassware: wavy patterns could be seen and also felt on the bottom of glass bowls, as if the glass had already start to soften. These marks are shown in the photo 2.

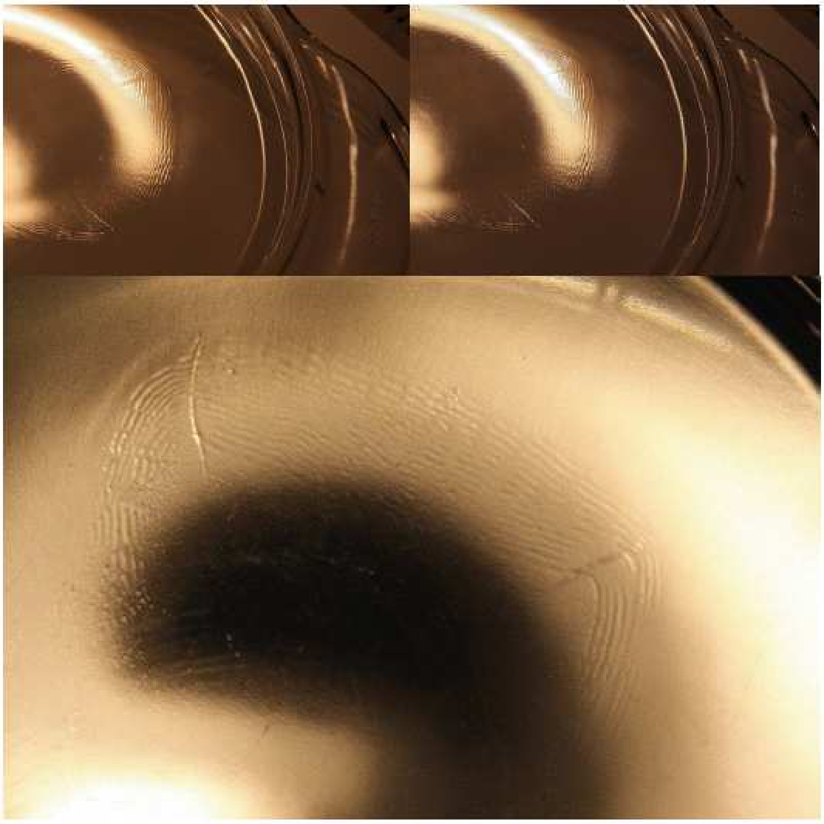


Photo 2. Wavy patterns on the bottom of a glass plate due to heating-cooling cycles and high temperatures.

As mentioned earlier, 350 °C was set as maximum temperature safety limit and 300 °C for average temperature. These limits should not be exceeded due to occupational safety reasons. In series 3 with 8/16 mm aggregates these two limits were constantly exceeded and therefore testing had to be broken off.

An interesting phenomenon was noticed when the 8/16 mm aggregate size was dried with the microwave oven, especially in series 3 (3200 W). Usually when high temperatures (> 350 °C) were measured, the entire sample was not that hot, but one or more “hot points” developed to tested sample, increasing also the average temperature. Temperature difference between these hot points and other parts of the sample mass was usually greater than 100 °C. After noticing the existence of hot points it was perceived that these points differed slightly from the other sample mass also by colour. Wet sample looked homogeneously grey, as well as the material dried with the reference method, but aggregate grains that got very hot during microwave oven drying turned reddish. From 500 g sample typically only one or two aggregate grains changed colour during heating. This colour difference can be seen from the photo 3.

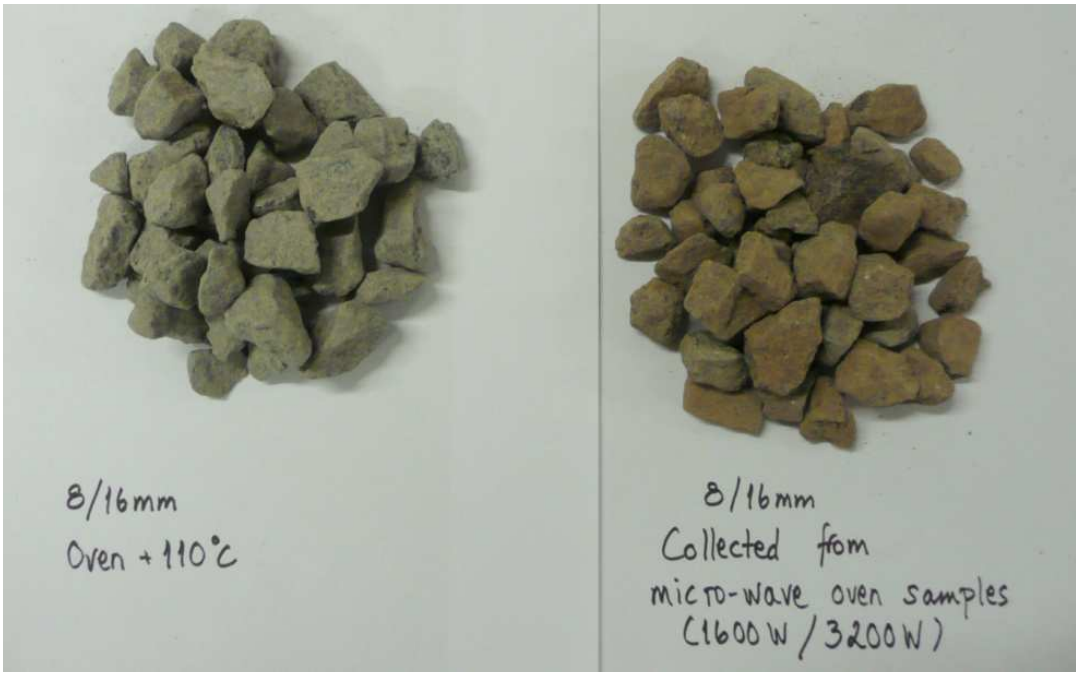


Photo 3. The colour difference between aggregates.

Otherwise these red aggregate grains looked similar to normal, grey grains. Some especially hot (> 400 °C) grains seemed to be porphyritic or even stratified but it cannot be taken as a separating factor since it was not the case with all, or even with the most of the hot aggregates. In the photo 4 two prophyritic-like aggregate grains with visible layer patterns are represented. Layers are indicated with arrows.

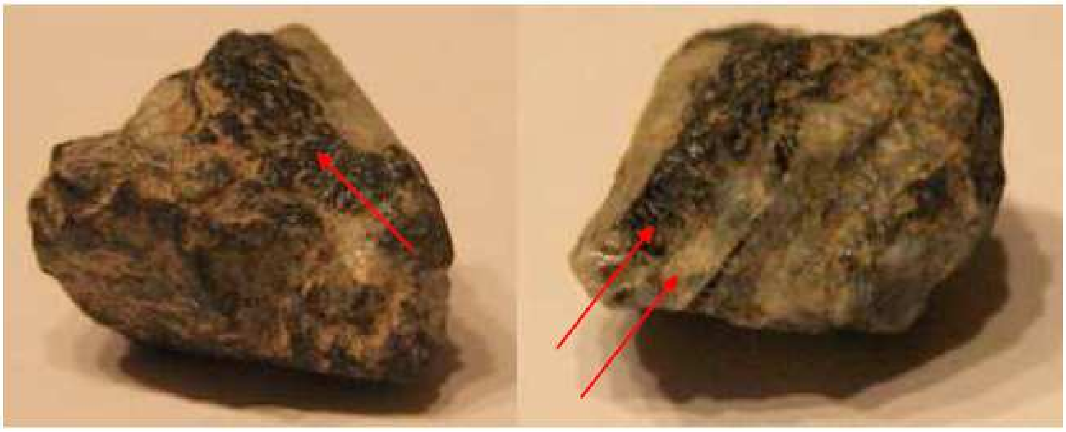


Photo 4. Porphyritic-like aggregate grains

Differences in aggregates were further tested with simple density tests: weighted amount of aggregates (m_{aggr}) was placed to exactly 500 ml (V_{water}) of water. New volume was recorded (V_{total}), and the volume ($V_{aggr} = V_{total} - V_{water}$) and the density of aggregates ($\rho_{aggr} = m_{aggr} / V_{aggr}$) was calculated. Results from red and normal aggregates can be seen from the table 24.

Table 24. Density difference with 8/16 mm.

Aggregate size 8/16 mm	**m_{aggr} [g]**	**V_{total} [dm^3]**	**V_{aggr} [dm^3]**	**ρ_{aggr} [kg/m^3]**
Red aggregates	196.0	0.570	0.070	**2800**
Normal aggregates	376.6	0.628	0.128	**2942**

Small difference in densities was noticed, but that can be taken only as an indicative result since collected sample mass was too small to be representative. For example the testing of particle density according to SFS EN 1097-6 it would require minimum 2 kg sample for 8/16 mm aggregates (SFS EN 1097-6, 2001, 9). Nevertheless, the density difference could suggest that there are different types of aggregates mixed in 8/16 mm, and one of these types reacts to microwave oven heating with rapid temperature increase. This is not proved or tested further in this study since it would require much more time than it was possible to use here.

Conclusions from heating time and power tests

The method tested in the series 3 (3200 W) was not considered for further tests due to the thermal behavior of 8/16 mm. Results obtained with methods tested in the series 1 (ASTM) and 2 (1600 W, long drying) did not truly differ from each other: small differences could be noticed, but from the overall view methods were quite even. Nevertheless the method tested in the series 2 was chosen for further stages. Testing was slightly easier with parameters tested in the series 2, when number of mixing and weighting times needed was smaller.

Chosen test parameters for the next stage are represented in the table 25.

Table 25. Test parameters chosen for the next stage.

Microwave oven power	1600 W
Heating sequences: 0/1 mm & 0/6 mm:	10 min + 1 min + 1 min + …
Heating sequences: 4/8 mm & 8/16 mm:	7 min + 1 min + 1 min + ...
Sample stirring before weighting	yes

7.3 Determination of optimum wet sample mass

Next the optimum wet sample mass was determined using the parameters in the table 25. Three test series with different sample mass, each consisting 10 samples, were carried out for each aggregate size. Samples mass for each aggregate size in each series are represented in the table 13. All moistures measured in this stage are represented in the table 26.

Table 26. Measured moistures.

Aggregate size [mm]	Sample	Reference		Series 1		Series 2		Series 3	
	number	Oven, hot [%]	Oven, cool [%]	Micro-wave, hot [%]	Micro-wave, cool [%]	Micro-wave, hot [%]	Micro-wave, cool [%]	Micro-wave, hot [%]	Micro-wave, cool [%]
0/1	1	9.5	**9.2**	9.3	9.0	9.6	9.2	9.4	9.1
	2	8.9	**8.6**	8.9	8.6	8.9	8.7	9.3	8.9
	3	9.5	**9.2**	9.4	9.0	9.5	9.2	9.5	9.2
	4	7.6	**7.2**	7.6	7.1	7.7	7.4	7.6	7.2
	5	7.8	**7.5**	8.0	7.5	7.9	7.5	7.7	7.4
	6	7.8	**7.5**	7.9	7.2	7.6	7.2	7.6	7.4
	7	9.2	**8.9**	9.7	9.2	9.1	8.8	9.3	8.9
	8	9.7	**9.3**	9.6	9.0	10.0	9.6	9.6	9.3
	9	9.4	**9.1**	9.3	8.7	9.4	9.0	9.4	9.1
	10	9.4	**9.2**	9.6	9.1	9.5	9.2	9.6	9.2
0/6	1	5.7	**5.2**	5.8	5.3	5.7	5.3	5.8	5.4
	2	4.9	**4.5**	5.2	4.8	5.1	4.7	5.3	4.9
	3	5.6	**5.2**	5.5	5.1	5.6	5.2	5.7	5.2
	4	4.8	**4.4**	4.9	4.5	4.8	4.3	4.8	4.3
	5	4.6	**4.2**	4.7	4.1	4.6	4.1	4.7	4.1
	6	5.4	**5.0**	5.4	4.9	5.4	4.9	5.4	4.8
	7	6.0	**5.7**	6.2	5.6	6.1	5.7	6.1	5.6
	8	5.7	**5.1**	5.9	5.3	5.8	5.3	5.7	5.0
	9	5.4	**5.1**	5.3	4.8	5.4	5.0	5.4	4.9
	10	5.4	**5**	5.3	4.9	5.2	4.9	5.4	4.9
4/8	1	2.6	**2.4**	2.6	2.5	2.7	2.6	3.0	2.8
	2	2.9	**2.7**	3.1	2.9	3.2	3.0	3.0	2.9
	3	2.9	**2.7**	3.0	2.8	2.7	2.6	2.8	2.6
	4	2.6	**2.5**	2.7	2.6	2.9	2.7	3.0	2.8
	5	2.9	**2.8**	2.8	2.6	2.8	2.6	2.9	2.7
	6	3.0	**2.9**	3.3	3.1	3.1	2.9	3.6	3.5
	7	3.3	**3.2**	3.0	2.9	3.3	3.1	3.3	3.2
	8	3.3	**3.1**	2.8	2.6	3.8	3.7	3.5	3.3
	9	2.8	**2.7**	2.7	2.5	2.8	2.6	2.7	2.6
	10	2.9	**2.8**	3.0	2.7	2.7	2.5	2.7	2.6
8/16	1	0.6	**0.5**	0.4	0.4	0.5	0.4	0.6	0.4
	2	0.6	**0.6**	0.6	0.6	0.7	0.7	0.7	0.6
	3	0.5	**0.5**	0.5	0.5	0.4	0.4	0.6	0.5
	4	0.8	**0.7**	0.6	0.6	0.8	0.7	0.9	0.8
	5	0.7	**0.6**	0.6	0.5	0.7	0.6	0.7	0.6
	6	0.7	**0.6**	0.7	0.6	0.4	0.3	0.6	0.5
	7	0.5	**0.4**	0.4	0.4	0.4	0.4	0.5	0.4
	8	0.8	**0.7**	0.7	0.7	0.8	0.8	0.7	0.6
	9	0.3	**0.2**	0.2	0.2	0.3	0.2	0.3	0.2
	10	0.5	**0.4**	0.4	0.3	0.3	0.2	0.4	0.3

Measured moistures for each aggregate size by series (the table 26) are represented in graphic form in the illustrations 18-21.

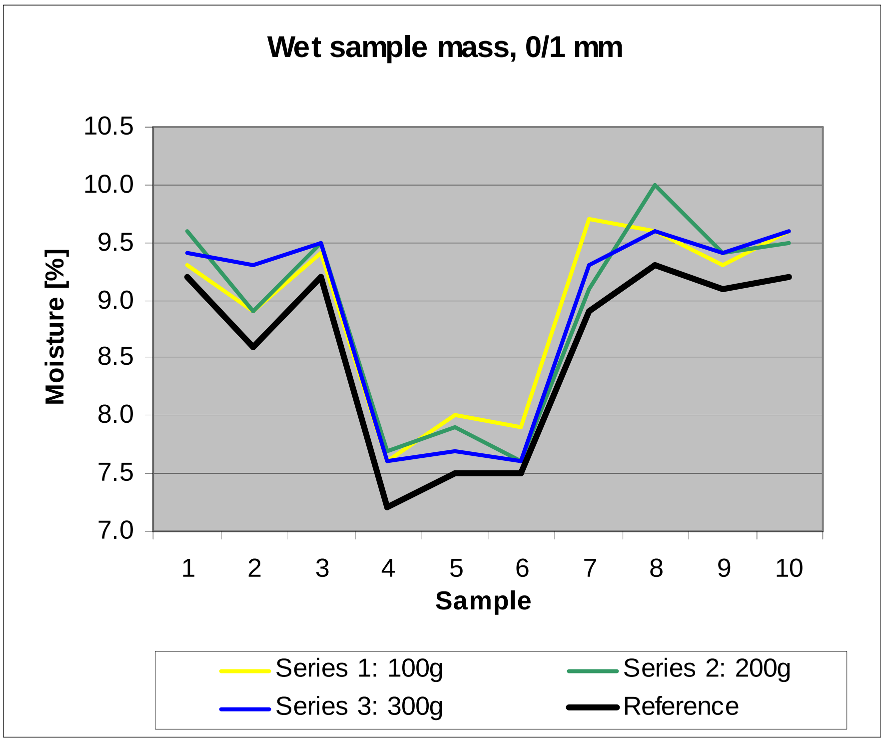


Illustration 18. Measured moistures, 0/1 mm.

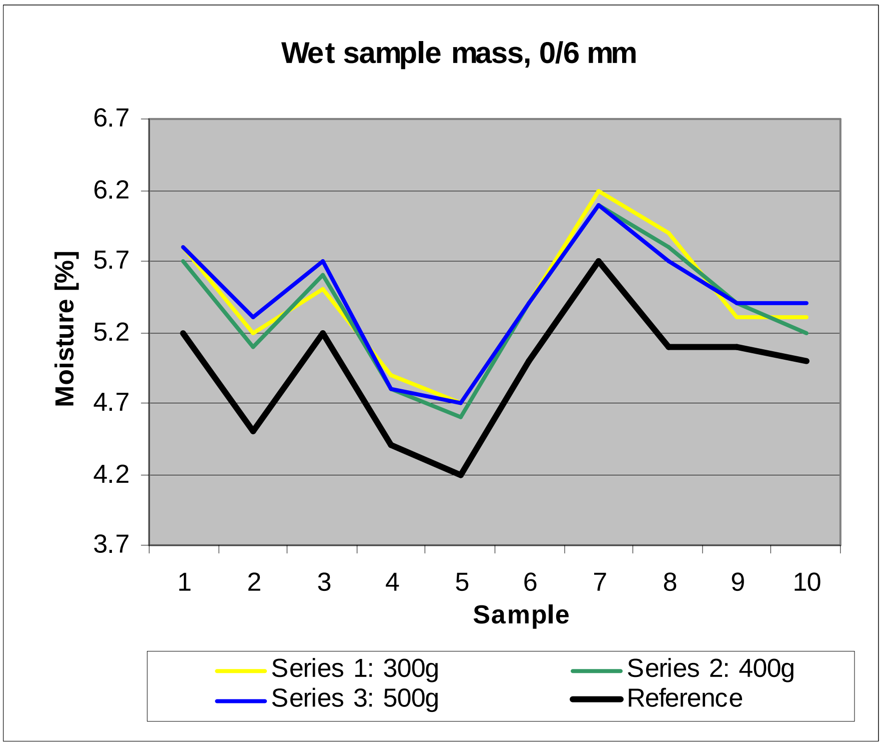


Illustration 19. Measured moistures, 0/6 mm.

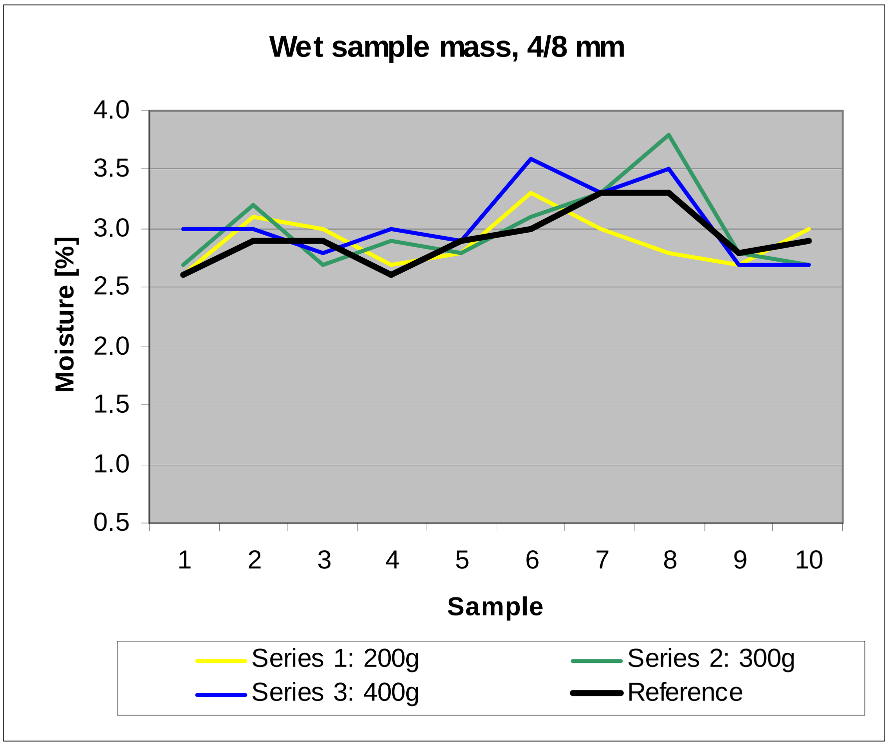


Illustration 20. Measured moistures, 4/8 mm.

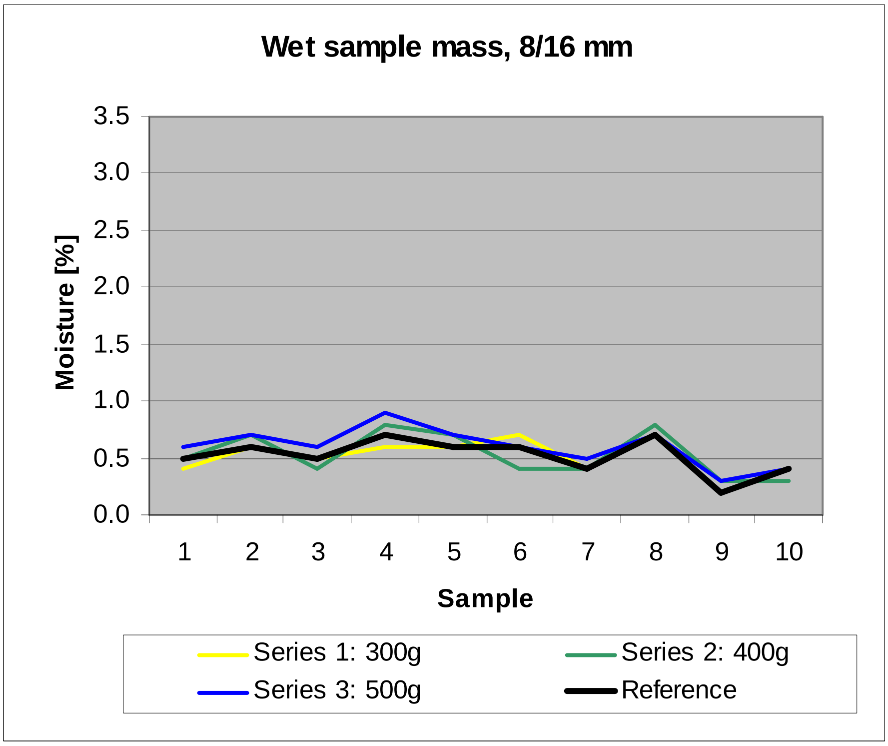


Illustration 21. Measured moistures, 8/16 mm.

The statistical analysis of the results is shown in the table 27. The series 1 includes the smallest wet sample size for each aggregate size, and the series 3 the biggest. Values for minimum, maximum and average as percentage units and also standard deviation are represented for moisture deviations between the reference method and the microwave oven method. Moistures measured with microwave oven were calculated using both: the hot sample weight and the weight of sample that has been allowed to cool down to room temperature.

Table 27. Statistical analysis of measured moistures.

		Series 1		**Series 2**		**Series 3**	
		reference microwave, hot	**reference microwave, cool**	**reference microwave, hot**	**reference microwave, cool**	**reference microwave, hot**	**reference microwave, cool**
0/1 mm	**Min**	0.1	-0.4	0.1	-0.3	0.1	-0.1
	Max	0.8	0.3	0.7	0.3	0.7	0.3
	Average	**0.4**	**-0.1**	**0.4**	**0.0**	**0.3**	**0.0**
	St.dev.	*0.20*	*0.20*	*0.16*	*0.17*	*0.16*	*0.10*
0/6 mm	**Min**	0.2	-0.3	0.2	-0.1	0.3	-0.2
	Max	0.8	0.3	0.7	0.2	0.8	0.4
	Average	**0.5**	**0.0**	**0.4**	**0.0**	**0.5**	**0.0**
	St.dev.	*0.19*	*0.18*	*0.14*	*0.12*	*0.14*	*0.19*
4/8 mm	**Min**	-0.3	-0.5	-0.1	-0.3	-0.1	-0.2
	Max	0.4	0.2	0.7	0.6	0.7	0.6
	Average	**0.1**	**-0.1**	**0.2**	**0.1**	**0.3**	**0.1**
	St.dev.	*0.24*	*0.24*	*0.25*	*0.27*	*0.27*	*0.26*
8/16 mm	**Min**	-0.1	-0.1	-0.2	-0.3	0.0	-0.1
	Max	0.1	0.0	0.1	0.1	0.2	0.1
	Average	**0.0**	**0.0**	**0.0**	**-0.1**	**0.1**	**0.0**
	St.dev.	*0.06*	*0.05*	*0.11*	*0.13*	*0.06*	*0.07*

With the results of 4/8 mm aggregates some difference between series occurs, but otherwise results are still quite even. Standard deviations within the results for one aggregate size are also relatively even; only 4/8 mm differs from the other. It is surprising to notice that its standard deviations are noticeably bigger than with other aggregate sizes.

Temperature

Temperature readings from this stage are represented in the table 28.

Table 28. Temperature readings.

Aggregate size [mm]	Sample	Series 1		Series 2		Series 3	
		Max temp.	Avg temp.	Max temp.	Avg temp.	Max temp.	Avg temp.
	number	[°C]	[°C]	[°C]	[°C]	[°C]	[°C]
0/1	1	109.2	92.9	101.8	82	85.8	71.6
	2	103.1	81.9	100.3	86.1	143.5	118.3
	3	89.1	77.3	92.4	78.9	117.3	93.1
	4	110.4	89.8	102.4	92	142.6	122
	5	109.5	87.4	113.6	89.8	130.6	110.9
	6	133.6	110.8	117.6	101	120.2	94.3
	7	112.9	88.6	99.3	87.3	128.3	103.2
	8	112.1	95.6	104.5	92.8	110.8	93.2
	9	121.2	98.7	109.4	101.2	118	98.2
	10	124.8	102.8	96.9	86.8	126.3	99.9
0/6	1	108	88.2	136	113.3	169.8	143.8
	2	167.9	134.9	116.7	104.4	148.2	127.7
	3	102.8	88.6	134.1	114.1	169.6	136.6
	4	172	138.9	120.3	102.3	147.8	122.9
	5	173.4	123.1	129	112.5	151.1	132.1
	6	133.3	105.9	131.8	107.6	119.6	105.9
	7	189.9	139.4	168.4	137.2	162.2	136.3
	8	167.6	135.4	183.3	154.5	161.5	140.1
	9	122.4	112.5	173.9	142.6	173.0	133.2
	10	138.7	120.4	123.4	105.1	157.8	142.1
4/8	1	117.6	87.3	120.9	88.9	182.4	130.7
	2	123.2	171.2	267.3	180.4	93.9	93.9
	3	125.6	93.6	137.6	102.7	189.3	153.6
	4	162.2	131.2	229.7	171.4	175.8	136.1
	5	209.9	169.3	186.5	158.2	188.8	129.6
	6	213.3	165.3	216.9	185.7	181.8	139.7
	7	154.1	127.3	237.5	187.5	229.8	181.8
	8	244.3	179.2	172.6	134.3	164.6	140.1
	9	214.2	150.2	215.3	146.3	217.8	149.8
	10	237.9	200.1	205.9	157.3	200.8	147.9
8/16	1	167.1	136.3	259.1	193	254.9	200.3
	2	193.6	136.9	360.5	248.4	303.2	247.7
	3	159.3	115.2	216.4	174	283.1	202.4
	4	178.3	144.7	493.7	319.8	314.8	227.1
	5	185.2	149.7	384.8	253.1	277.4	185.8
	6	272.8	209	269.4	194.9	346.7	245.7
	7	176	150.3	237.8	187.7	221.1	182.5
	8	219.5	161	321.9	240.6	227	165.9
	9	216.1	147.4	288.1	199.2	278.6	215.7

As noticed in the chapter 7.2 the only aggregate size that can cause a temperature risk is 8/16 mm. Data in the table 28 supports this observation, and in the illustrations 22 and 23 the maximum and average temperatures from 8/16 mm by test series are represented in graphic form, respectively.

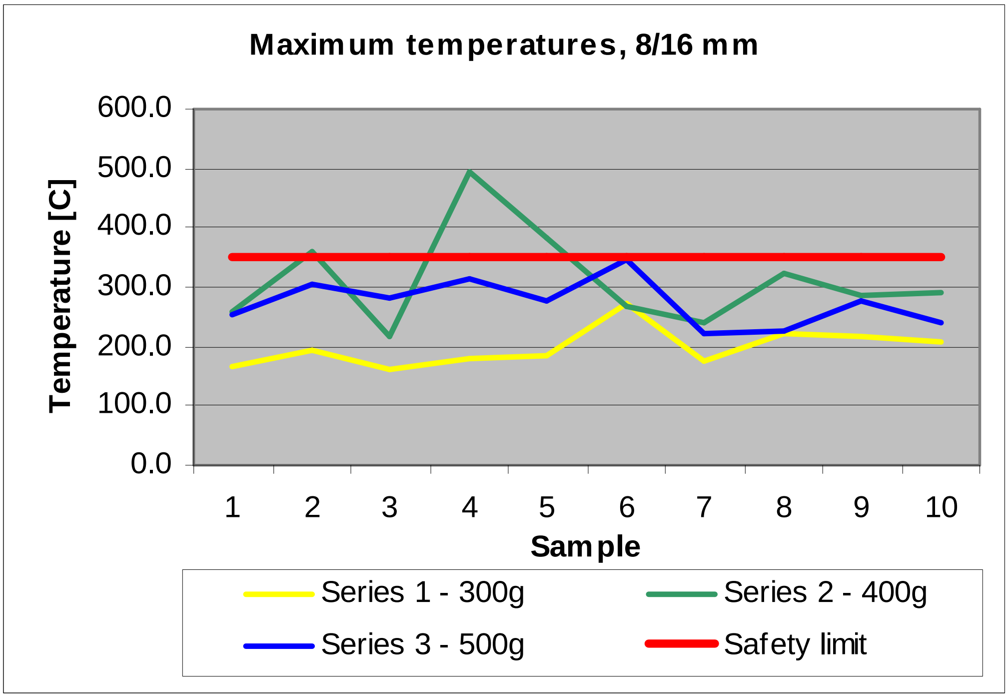


Illustration 22. Maximum temperatures, 8/16 mm.

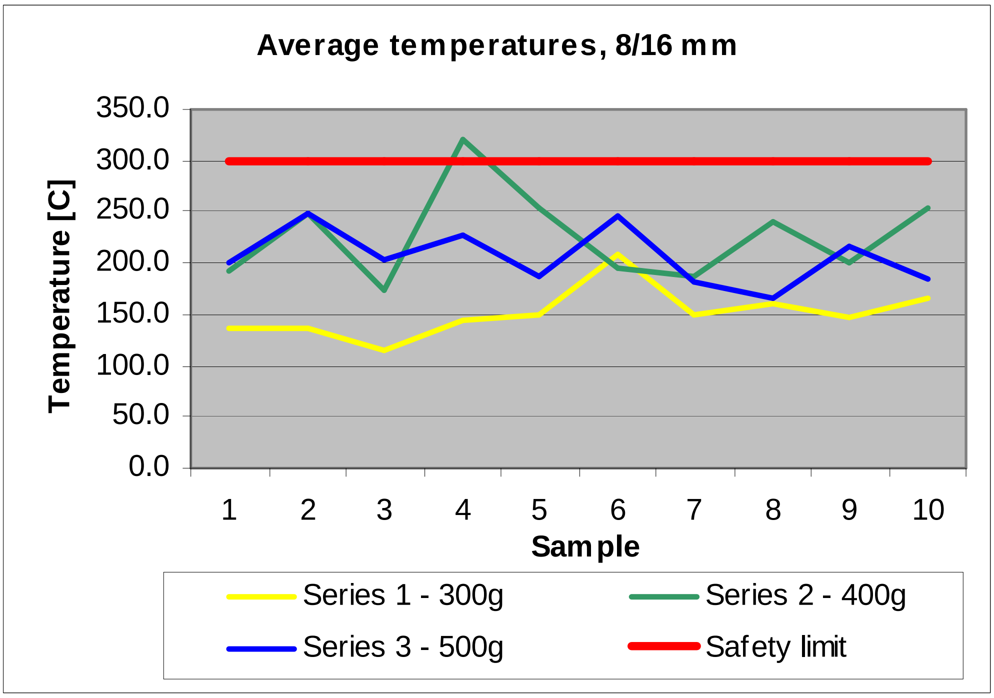
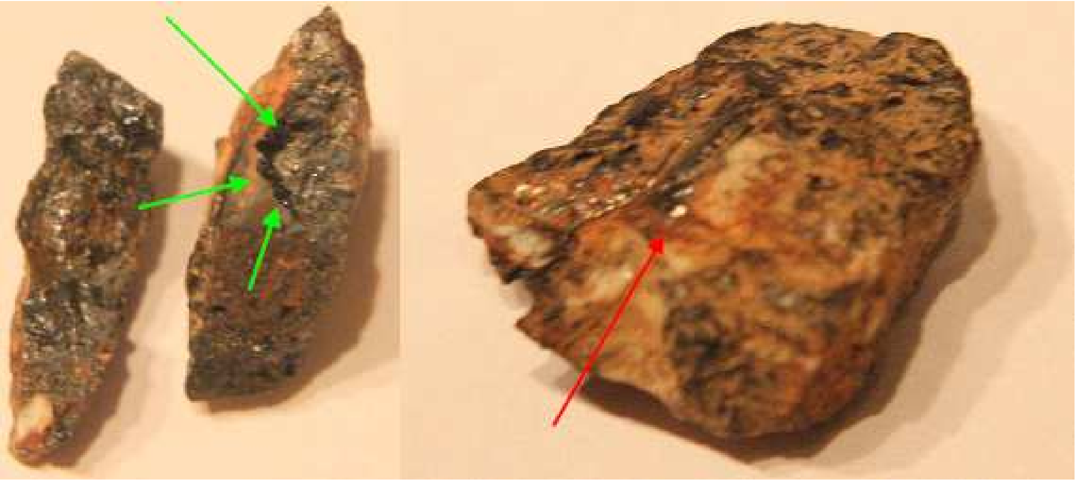


Illustration 23. Average temperatures, 8/16 mm.

From the pictures 22 and 23 it can be seen that extremely high temperatures were measured from the sample 4 in the series 2. The hot point was clearly focused in only one aggregate grain, and the average temperature of that sample confirms this: if the sample was thoroughly too hot, also the average temperature would be significantly over the safety limit, as it was the case in the stage 2. Now the average temperature safety limit is exceeded only by 20 °C. Since there was only one alarming temperature measurement during this stage it was taken as an individual exception.

During the test the hot point could also be seen: when the sample was taken out from the microwave oven this one grain had glowing red point; like metal when molten. The maximum temperature was measured from this glowing point. After the sample was cooled down, this particular grain was examined: a small crack on the grain was noticed, and in the bottom of this crack was a small golden-colored chip. This crack was clearly the origin of the glow, since the crack and its surroundings were turned to black. This grain is shown in the photo 5, but unfortunately the equipment was not very suitable for the target and photos do not have very good quality. The green arrows in the left-hand side photo indicate the small crack, and the red arrow on the right-hand side photo the golden-colored chip.



Picture 5. The grain that developed glowing hot point during microwave oven heating.

This golden-colored chip and its dramatic reaction to microwave oven heating gave reason to doubt that there might be some metal traces or impurities inside aggregates.

Conclusions of determination of optimum wet sample mass

The sample sizes for further testing are chosen according to the smallest average difference between the moistures measured with the reference method and the microwave oven (table 27). Therefore sample sizes chosen for the final stage are as follows represented in the table 29.

Table 29. Mass of wet sample for the final stage.

Aggregate size]mm]	**Mass of wet sample [g]**
0/1	300
0/6	400
4/8	200
8/16	300

7.4 Simplified procedure

Final aim was to simplify the testing procedure: it was done by checking the influence of stirring on the aggregate sample. 10 samples for each aggregate size were tested. Wet sample was heated in the microwave oven only once and the dry sample was weighed without stirring. Measured moistures are represented in the table 30.

Table 30. Measured moistures

Aggregate size [mm]	Sample	Reference		Part 4	
	number	Oven, hot [%]	Oven, cool [%]	Microwave, hot [%]	Microwave, cool [%]
0/1	1	9.5	**9.0**	9.3	9.0
	2	9.4	**9.0**	8.7	9.0
	3	8.7	**8.5**	8.6	8.3
	4	9.3	**9.1**	8.9	9.0
	5	8.2	**7.9**	8.3	7.9
	6	9.1	**8.8**	9.1	8.7
	7	9.5	**9.3**	9.5	9.1
	8	9.0	**8.8**	9.1	8.7
	9	8.3	**8.0**	7.9	8.0
	10	9.4	**9.1**	9.5	9.2
0/6	1	6.3	**5.9**	6.1	5.6
	2	5.6	**5.2**	5.6	5.1
	3	5.5	**5.2**	5.3	4.9
	4	5.5	**5.3**	5.7	5.1
	5	5.9	**5.5**	5.8	5.3
	6	5.7	**5.4**	5.6	5.2
	7	5.9	**5.6**	5.7	5.3
	8	5.7	**5.4**	5.5	5.2
	9	5.8	**5.5**	5.8	5.4
	10	6.1	**5.6**	5.7	5.5
4/8	1	3.0	**1.7**	2.1	1.6
	2	2.2	**2.4**	2.7	2.6
	3	3.3	**2.3**	2.4	2.2
	4	3.2	**2.5**	2.7	2.5
	5	3.1	**2.4**	2.9	2.6
	6	2.7	**2.2**	2.7	2.3
	7	2.8	**1.8**	2.4	2.1
	8	2.8	**2.3**	2.6	2.3
	9	3.1	**2.5**	2.9	2.7
	10	2.5	**2.4**	2.8	2.6
8/16	1	0.7	**0.6**	0.6	0.6
	2	0.5	**0.4**	0.5	0.4
	3	0.6	**0.5**	0.4	0.4
	4	0.5	**0.5**	0.5	0.4
	5	0.6	**0.6**	0.6	0.5
	6	0.6	**0.6**	0.6	0.5
	7	0.6	**0.6**	0.5	0.5
	8	0.5	**0.4**	0.5	0.4
	9	0.8	**0.6**	0.6	0.6
	10	0.6	**0.6**	0.5	0.4

Results obtained with 4/8 mm were not satisfactory, and the testing of 4/8 mm was continued with two other series. In the series 2 the wet sample mass was increased to 400 g, which was the maximum wet sample mass tested in the stage 3. Otherwise the procedure was kept the same. In the series 3 the original wet sample mass tested in this stage, 200 g, was used, and the sample was dried in the microwave oven only once, but the dry sample was mixed with spatula before weighing. 10 samples were tested also for the series 2 and 3. All samples were weighed after cooling down as well. Measured moistures obtained in the series 2 and 3 are represented in the table 31.

Table 31. Measured moistures, 4/8 mm, series 2 and 3,

Aggregate size [mm]	**Sample**	**Reference**		**Stage 4**	
	number	**Oven, hot [%]**	**Oven, cool [%]**	**Microwave, hot [%]**	**Microwave, cool [%]**
4/8, series 2	**11**	2.7	**2.5**	2.9	2.7
	12	2.5	**2.4**	2.8	2.7
	13	3.1	**3.0**	3.3	3.0
	14	3.2	**3.1**	3.2	3.0
	15	3.3	**3.2**	3.5	3.3
	16	2.7	**2.5**	2.9	2.7
	17	2.8	**2.6**	2.9	2.6
	18	3.1	**2.9**	3.3	3.2
	19	2.4	**2.3**	2.4	2.7
	20	3.0	**2.9**	2.8	2.5
4/8, series 3	**21**	3.0	**2.9**	2.7	2.5
	22	2.2	**2.0**	2.3	2.0
	23	3.3	**3.1**	3.1	2.8
	24	3.2	**3.0**	3.3	3.0
	25	3.1	**3.0**	3.1	2.9
	26	2.7	**2.6**	2.8	2.6
	27	2.8	**2.7**	2.8	2.7
	28	2.8	**2.7**	2.6	2.5
	29	3.1	**3.0**	2.8	2.8
	30	2.5	**2.4**	2.2	2.1

Moisture results from the table 30 for each aggregate size by series are represented in graphic form in the illustrations 24-27.

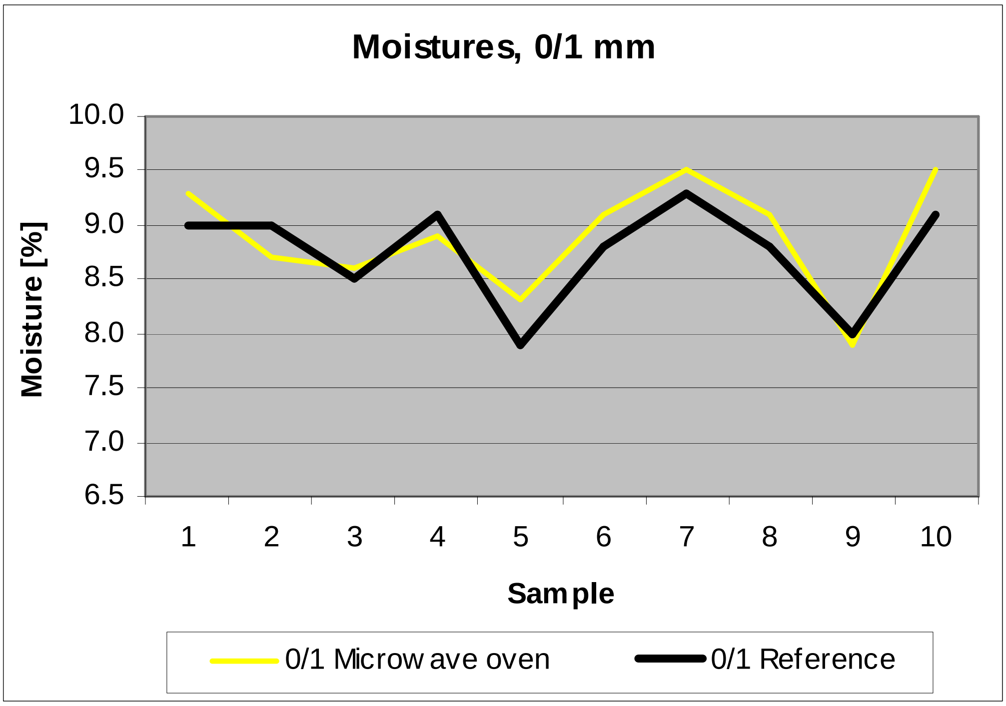


Illustration 24. Measured moistures, 0/1 mm.

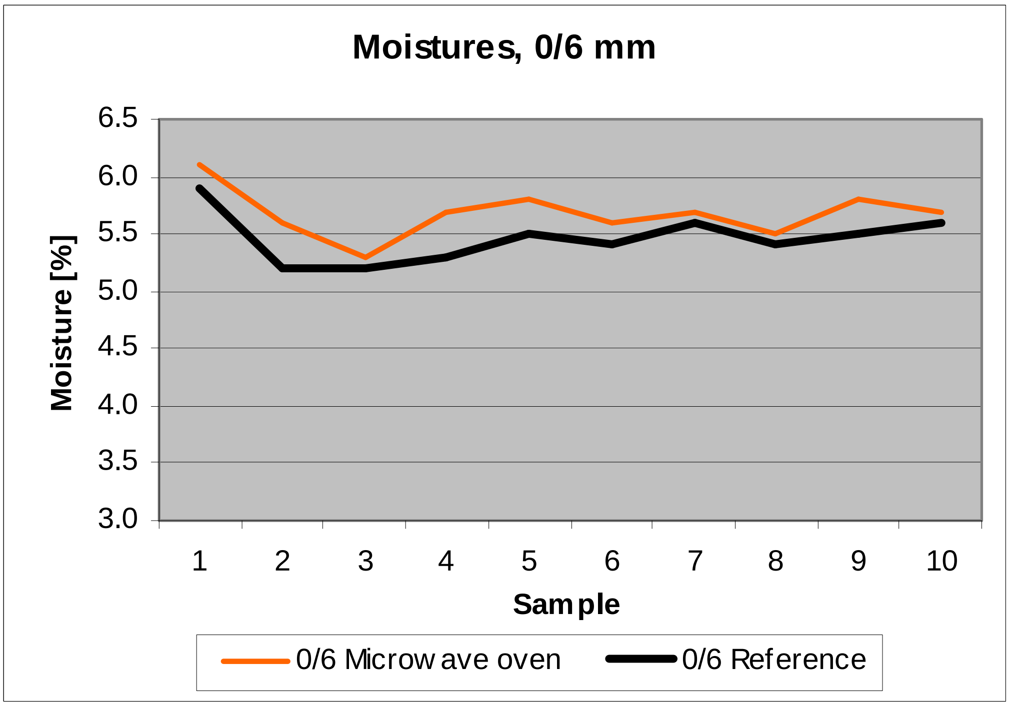


Illustration 25. Measured moistures, 0/6 mm.

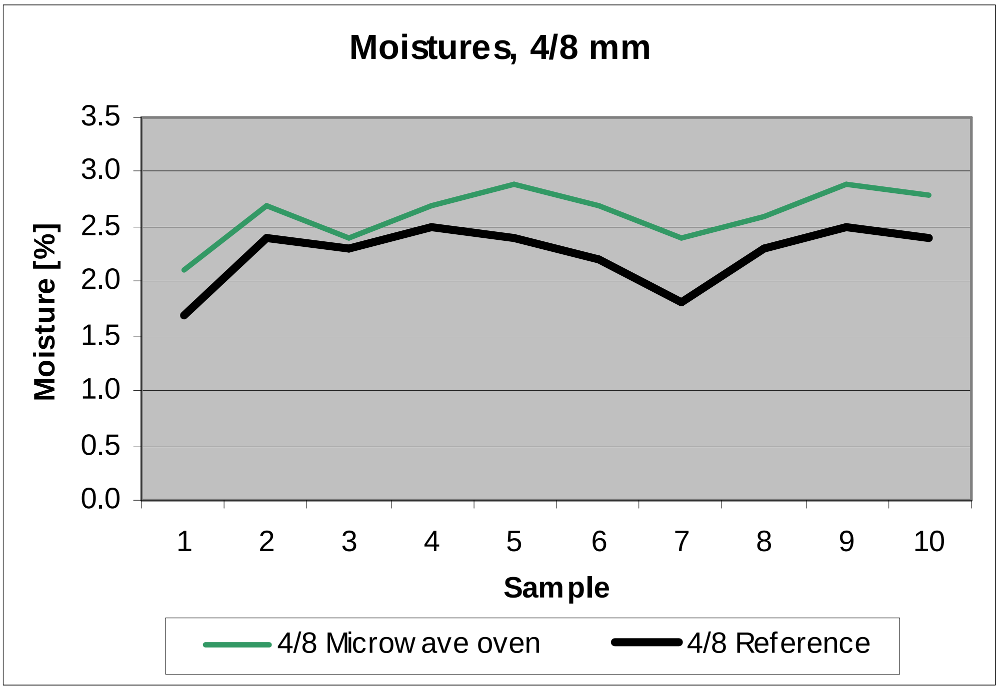


Illustration 26. Measured moistures, 4/8 mm.

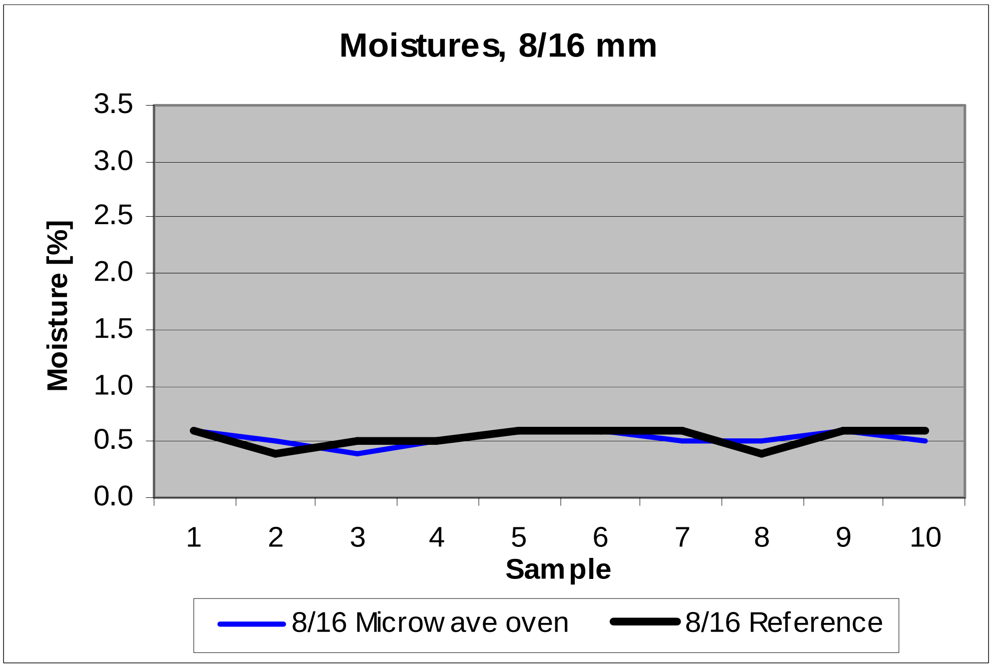


Illustration 27. Measured moistures, 8/16 mm.

Statistical analysis from the results is represented in the table 32.

Table 32. Statistical analysis of measured moistures.

		microwave, hot	microwave, cool	reference microwave, hot	reference microwave, cool
0/1 mm	**Min**	7,9	7,9	-0,3	-0,2
	Max	9,5	9,2	0,4	0,1
	Average	**8,9**	**8,7**	**0,1**	**-0,1**
	St.dev.	*0,52*	*0,47*	*0,25*	*0,10*
0/6 mm	**Min**	5,3	4,9	0,1	-0,3
	Max	6,1	5,6	0,4	-0,1
	Average	**5,7**	**5,3**	**0,2**	**-0,2**
	St.dev.	*0,21*	*0,21*	*0,12*	*0,08*
4/8 mm	**Min**	2.1	1.6	0.1	-0.1
	Max	2.9	2.7	0.6	0.3
	Average	**2.6**	**2.4**	**0.4**	**0.1**
	St.dev.	*0.25*	*0.33*	*0.15*	*0.14*
8/16 mm	**Min**	0.4	0.4	-0.1	-0.2
	Max	0.6	0.6	0.1	0.0
	Average	**0.5**	**0.5**	**0.0**	**-0.1**
	St.dev.	*0.07*	*0.08*	*0.07*	*0.07*

In the table 33 the best series for each aggregate size from the previous stage (table 27) are compared to data from the table 32.

Table 33. Comparison of stages 3 and 4.

		Stage 3		Stage 4
		reference microwave, hot		reference microwave, hot
0/1 mm	**Average**	0,3	>	0,1
	St.dev.	*0,16*		*0,25*
0/6 mm	**Average**	0,4	>	0,2
	St.dev.	*0,14*		*0,12*
4/8 mm	**Average**	0,1	<	0,4
	St.dev.	*0,24*		*0,15*
8/16 mm	**Average**	0,0	=	0,0
	St.dev.	*0,06*		*0,07*

The table 33 shows that tested method was improved and results are better for all other aggregate sizes except for 4/8mm. Results of 8/16 mm are in both parts as good as they can possibly get, and 0/1 mm and 0/6 mm have improved by 50 % or more. The tendency is not the same with 4/8 mm: results remained better in the part 3. Since the method that does not include mixing worked well with other aggregate sizes, next thought was that the conclusion made from the part 3 was not correct after all and the sample size was not big enough for 4/8 mm. The next series for 4/8 mm was carried out with maximum wet sample size tested in the part 3, 400 g. Otherwise the method was kept the same. The statistical analysis of this series is represented in the table 34.

Table 34. Series 2: statistical analysis of measured moistures.

		Series 2			
		microwave, hot	**microwave, cool**	**reference microwave, hot**	**reference microwave, cool**
4/8, 400 g	**Min**	2,4	2,5	-0,1	-0,4
	Max	3,5	3,3	0,4	0,4
	Average	**3,0**	**2,8**	**0,3**	**0,1**
	St.dev.	*0,32*	*0,27*	*0,17*	*0,24*

Results did not improve in this series, contrary the average difference between reference method and hot sample tested with the microwave oven method increased. It was clear that path that was followed through stages 1, 2 and 3 was right and the answer for improving the results had to be found from somewhere else. The one thing that was changed between last two stages was the mixing; therefore in series 3 the method was changed. The wet sample size was kept the same than in series 1 (200 g), and the sample was heated in the microwave oven once but before weighing it was mixed with spatula. The statistical analysis from the series 4 is represented in the table 35.

Table 35. Series 3: statistical analysis of measured moistures.

		Series 3			
		microwave, hot	**microwave, cool**	**reference / microwave, hot**	**reference / microwave, cool**
4/8, 200 g	**Min**	2,2	2,0	-0,2	-0,4
	Max	3,3	3,0	0,3	0,0
	Average	**2,8**	**2,6**	**0,0**	**-0,3**
	St.dev.	*0,35*	*0,33*	*0,20*	*0,15*

Results from the table 35 are well in line with results from other aggregate sizes in the table 32. It is clear that 4/8 mm aggregate size needs stirring in the microwave oven drying unlike other aggregate sizes.

Conclusion of the simplification of the test

Stirring of the aggregate sample during microwave oven drying is not necessary for 0/1 mm, 0/6 mm and 8/16 mm aggregate fractions. Weighing of the dry sample without mixing gave approximately 50 % less difference compared to the reference method, than did the drying procedure with mixing. Surprisingly mixing was needed for 4/8 mm aggregates. The method was still simplified and results improved compared to previous parts.

8 CONCLUSIONS

After this study it is obvious that the determination of aggregate moisture content using the microwave oven method suits well for the use of concrete production. As presented in the table 36 the microwave oven method has been proved to be

- Significantly quicker than other gravimetric methods, such as the standardized ventilated oven or the gas burner method
- More accurate and reliable than the gas burner method which is usually used on other building sites
- More easy and light to work with. Any special arrangements, such as gas bottles or ventilation, are not needed and all equipments are small, cheap can easily be found from the market.

Table 36. Final comparison of gravimetric methods.

		Reference method	**Gas burner method**	**Microwave oven method**
Test duration		20 h	45 min	12 min
Difference in moisture content compared to the reference method [percentage unit]	**0/1**	-	1,0	0,1
	0/6 0/8	-	0,9	0,2
	4/8	-	not tested	0,0
	8/16	-	0,3	0,0

In order to show the impact of different drying methods to the efficient water amount in concrete production, an example is illustrated in the table 37. The most used concrete mix design in OL3 project contains 185 liters of effective water per cubic meter of concrete. Aggregate amounts used for 1 m^3 of this concrete are shown in the table 37 in addition to water amounts coming from aggregates using each moisture determination method.

Table 37. An example of the impact of the moisture determination method to the total water amount in cubic meter of concrete.

Aggregate size [mm]	**Mass of each aggregate size [kg] used for 1 m^3 of concrete**	**Moisture [%], measured with**			**Calculative amount of water [kg] from aggregates to 1 m^3 of concrete , measured with**		
		Reference	Gas burner	Microwave oven	Reference	Gas burner	Microwave oven
0/1	145	9.0	10.0	9.1	13.1	14.5	13.2
0/6	791	5.0	5.9	5.2	39.6	46.7	41.1
4/8	310	3.0	3.4	3.0	9.3	10.5	9.3
8/16	681	0.6	0.9	0.6	4.1	6.1	4.1
TOTAL AMOUNT OF WATER [kg] FROM AGGREGATES, calculated using each method					**66.1**	**77.8**	**67.7**
Difference to the reference method [kg]					**-**	**11.7**	**1.6**
Percentage of total amount of water needed (185 kg/m^3) [%]					**35.7**	**42.1**	**36.6**

The difference in total water amounts between the gas burner method and the microwave oven method is remarkable and its effect to workability of concrete would be significant. As seen from the table 37 the gas burner method by itself will overestimate the quantity of efficient water with more than 10 liters per cubic meter of concrete. To achieve the same consistency this amount of water has to be added to the concrete mix, and although the actual effective water amount in the concrete mix is correct, 185 l/m^3, the calculative water-cement ratio appears to be approximately 0,03 units too high. Thus the overestimation of water from the aggregates leads also to overestimation of the calculative water-cement ratio.

The most suitable testing procedure for the microwave oven drying was determined. The parameters for the optimized procedure are represented in the table 38.

Table 38. The procedure for the microwave oven method.

Aggregate size [mm]	**0/1**	**0/6**	**4/8**	**8/16**
Wet sample size [g]	300	400	200	300
Microwave oven power [W]	1600	1600	1600	1600
Time in the oven [min]	10	10	7	7
Stirring before weighing	no	no	yes	no
Total test duration [min]	12,5	12,5	9	8,5

With coarse aggregates temperature increase can be remarkable, and therefore hot samples have to be handled with care and using protective gloves, and dishes used for testing need to be allowed to cool down without causing thermal shock to them.

All these tests and this thesis were realized in the OL3 project in Olkiluoto. Determination of the moisture content of aggregates with microwave oven has been noticed to be very useful in the concrete production, and in the OL3 project this method is used daily.

It has been noticed by the batching plant operators and the concrete laboratory, that the microwave method offers quick and reliable results. This aspect has been really observed when the concrete consistency has drastically changed during continuous concrete production. Mainly, this change of consistency is due to the fact that aggregates from a new delivery with different moisture content are used. Also during wintertime when the aggregates are heated in order to get warm concrete, the aggregate mass in storage or a silo can locally differ in temperature and moisture content affecting the workability of the concrete. With this microwave oven method, new moisture contents are defined after only one, or maximum two trucks produced in the continuous production which allows maintaining preferred consistency and realistic water-cement ratios for all concrete mix designs at all times.

Since good results with microwave oven drying of aggregates were obtained, a future perspective could be the determination of water content of fresh concrete by the microwave oven method. The water content of fresh concrete could be checked easily and quickly, and the method could give important quality information to the concrete production for example in the case when concrete workability changes unexpectedly.

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APPENDICES

Appendix 1, page 1: Photos of equipment used for the thesis

Photo 1: Equipment used for the reference method.

Photo 2: Equipment used for the microwave oven method.

Photo 3: Equipment used for the microwave oven method.

Photo 4: Equipment used for temperature measurements.

Appendix 2: Measured moistures with the reference method, gas burner method and microwave oven method (NF P94-049-1)

Test number	Reference method				Gas burner method				Microwave oven method (NF P94-049-1)			
	0/1	0/8		8/16	0/1	0/8		8/16	0/1	0/8		8/16
Test 01	7.5%	4.2%	4.3%	0.5%	8.7%	5.0%	5.1%	0.8%	7.8%	4.5%	4.6%	-
Test 02	7.8%	5.5%	5.5%	0.9%	8.6%	6.3%	6.3%	1.1%	8.1%	5.9%	5.9%	0.9%
Test 03	9.0%	5.7%	5.6%	1.1%	10.1%	6.7%	6.6%	1.4%	9.3%	6.4%	6.1%	1.3%
Test 04	8.4%	4.8%		0.9%	9.5%	5.7%		1.4%	8.9%	5.6%		1.1%
Test 05	8.5%	4.7%		0.9%	9.4%	5.6%		1.2%	9.0%	5.3%		
Test 06	9.0%	3.9%	3.8%	0.6%	10.1%	4.8%	4.4%	1.0%	9.6%	4.4%	4.1%	0.8%
Test 07	9.2%	5.9%	5.9%	0.9%	10.3%	6.6%	6.5%	1.1%	9.3%	6.3%	6.0%	0.9%
Test 08	9.2%	5.5%	5.4%	1.1%	10.1%	6.3%	6.2%	1.4%	9.2%	6.1%	5.6%	1.2%
Test 09	8.8%	5.4%	5.3%	1.1%	10.0%	6.2%	6.2%	1.3%	9.2%	5.8%	5.6%	1.2%
Test 10	7.6%	5.0%		0.7%	8.8%	6.1%		1.0%	8.2%	5.6%		-
Test 11	7.8%	4.6%		0.7%	8.9%	5.8%		1.0%	8.0%	-		0.8%
Test 12	8.1%	4.8%		0.5%	8.9%	5.9%		1.0%	8.3%	5.0%		0.7%
Test 13	8.5%	4.9%		0.4%	9.7%	6.0%		0.8%	8.4%	-		0.6%
Test 14	7.9%	5.0%	4.9%	0.6%	9.1%	6.0%	5.9%	1.0%		5.2%		0.8%
Test 15	7.8%	4.8%	4.7%	0.6%	8.9%	5.8%	5.7%	1.1%	8.5%	5.5%	5.3%	0.8%
Test 16	9.0%	4.5%		-	10.4%	5.7%		-	9.6%	5.1%		-
Test 17	7.9%	4.9%		-	9.1%	5.7%		-	8.3%	5.2%		-
Test 18	8.2%	4.7%		0.5%	9.3%	5.6%		1.0%	8.6%	5.0%		0.7%
Test 19	7.3%	4.3%		0.7%	8.7%	5.1%		1.0%	8.0%	4.8%		0.8%
Test 20	7.2%	5.4%		0.4%	8.5%	6.1%		0.7%	7.8%	5.6%		0.5%
Test 21	7.3%	4.4%		0.1%	8.6%	5.5%		0.6%	8.1%	5.0%		0.3%
Test 22	7.3%	4.7%		0.6%	8.3%	5.7%		0.6%	8.1%	5.0%		0.6%

Appendix 3:Measured moistures with the reference method, gas burner method and microwave oven method (ASTM D 4643-08)

Test number	Reference method			Gas burner method			Microwave oven method (ASTM D 4643-08)		
	0/1	**0/8**	**8/16**	**0/1**	**0/8**	**8/16**	**0/1**	**0/8**	**8/16**
Test 21	7.9%	5.2%	0.6%	8.8%	6.1%	0.8%	8.0%	5.4%	0.6%
Test 22	7.9%	5.4%	0.6%	8.9%	6.2%	0.8%	8.3%	5.5%	0.6%
Test 23	8.1%	6.1%	1.1%	9.1%	7.0%	1.2%	8.3%	6.5%	1.0%
Test 24	8.1%	6.1%	-	9.1%	7.2%	-	8.6%	6.6%	-
Test 25	8.1%	4.8%	1.3%	8.9%	5.5%	1.7%	8.6%	4.8%	1.4%
Test 26	-	6.1%	0.7%	-	6.9%	1.1%	-	6.6%	1.1%
Test 27	8.1%	5.6%	0.9%	9.0%	6.5%	0.9%	8.4%	5.4%	0.7%
Test 28	9.4%	4.7%	1.1%	10.2%	5.5%	1.3%	9.8%	4.8%	1.2%
Test 29	9.2%	4.6%	1.1%	10.2%	5.4%	1.4%	9.4%	4.9%	1.2%
Test 30	9.2%	5.3%	1.0%	10.1%	6.0%	0.9%	9.9%	5.4%	1.1%
Test 31	9.1%	4.9%	1.1%	9.9%	5.4%	1.3%	9.5%	5.1%	1.2%
Test 32	9.2%	4.6%	1.3%	10.1%	5.6%	1.6%	9.4%	5.0%	1.2%
Test 33	8.4%	5.0%	1.2%	9.5%	5.9%	1.5%	8.9%	5.4%	1.2%
Test 34	9.0%	5.8%	1.3%	9.8%	6.8%	1.8%	9.6%	6.1%	1.6%
Test 35	9.1%	6.3%	1.2%	10.2%	6.9%	1.5%	9.6%	6.1%	1.2%
Test 36	9.2%	6.2%	1.2%	9.8%	7.0%	1.5%	9.5%	6.3%	1.1%
Test 37	8.0%	5.7%	1.0%	8.8%	6.5%	1.3%	7.8%	5.9%	1.0%
Test 38	7.5%	4.6%	0.7%	8.4%	5.5%	1.3%	7.5%	4.9%	0.9%
Test 39	7.5%	4.7%	0.9%	8.7%	5.9%	1.3%	7.8%	5.1%	1.1%
Test 40	7.5%	4.8%	0.9%	8.6%	6.0%	1.2%	7.8%	5.2%	0.8%
Test 41	8.3%	4.9%	0.1%	9.6%	5.9%	0.5%	8.8%	5.3%	0.3%
Test 42	9.0%	5.0%	0.3%	10.0%	5.9%	0.8%	9.4%	4.8%	0.4%
Test 43	7.9%	4.8%	0.7%	8.7%	5.8%	0.8%	8.1%	5.2%	0.6%
Test 44	8.1%	4.8%	0.5%	9.1%	5.9%	0.7%	8.3%	5.2%	0.5%
Test 45	8.5%	4.7%	0.4%	9.2%	5.5%	0.6%	9.4%	4.9%	0.4%
Test 46	7.9%	5.2%	0.2%	8.5%	5.8%	0.7%	8.2%	5.2%	0.1%
Test 47	8.5%	5.4%	-	10.6%	6.1%	1.1%	9.1%	5.5%	-
Test 48	7.9%	4.7%	-	9.2%	5.9%	0.9%	8.5%	5.3%	-

Appendix 4: Differences in measured moisture contents between tested drying methods (the gas burner method and the microwave oven methods, NF P94-049-1 and ASTM D 4643-08) compared to the reference method as percentage units.

	0/1		0/8		8/16	
	ref burners	ref m.wave (ASTM)	ref burners	ref m.wave (ASTM)	ref burners	ref m.wave (ASTM)
Test 21	0.9	0.1	0.9	0.2	0.2	0.0
Test 22	1.0	0.4	0.8	0.1	0.2	0.0
Test 23	1.0	0.2	0.9	0.4	0.1	-0.1
Test 24	1.0	0.5	1.1	0.5		
Test 25	0.8	0.5	0.7	0.0	0.4	0.1
Test 26			0.8	0.5	0.4	0.4
Test 27	0.9	0.3	0.9	-0.2	0.0	-0.2
Test 28	0.8	0.4	0.8	0.1	0.2	0.1
Test 29	1.0	0.2	0.8	0.3	0.3	0.1
Test 30	0.9	0.7	0.7	0.1	-0.1	0.1
Test 31	0.8	0.4	0.5	0.2	0.2	0.1
Test 32	0.9	0.2	1.0	0.4	0.3	-0.1
Test 33	1.1	0.5	0.9	0.4	0.3	0.0
Test 34	0.8	0.6	1.0	0.3	0.5	0.3
Test 35	1.1	0.5	0.6	-0.2	0.3	0.0
Test 36	0.6	0.3	0.8	0.1	0.3	-0.1
Test 37	0.8	-0.2	0.8	0.2	0.3	0.0
Test 38	0.9	0.0	0.9	0.3	0.6	0.2
Test 39	1.2	0.3	1.2	0.4	0.4	0.2
Test 40	1.1	0.3	1.2	0.4	0.3	-0.1
Test 41	1.3	0.5	1.0	0.4	0.4	0.2
Test 42	1.0	0.4	0.9	-0.2	0.5	0.1
Test 43	0.8	0.2	1.0	0.4	0.1	-0.1
Test 44	1.0	0.2	1.1	0.4	0.2	0.0
Test 45	0.7	0.9	0.8	0.2	0.2	0.0
Test 46	0.6	0.3	0.6	0.0	0.5	-0.1
Test 47	2.1	0.6	0.7	0.1		
Test 48	1.3	0.6	1.2	0.6		

<table>
<tr><th></th><th colspan="2">0/1</th><th colspan="4">0/8</th><th colspan="2">8/16</th></tr>
<tr><th></th><th>ref burners</th><th>ref m.wave (NFP)</th><th colspan="2">ref burners</th><th colspan="2">ref m.wave (NFP)</th><th>ref burners</th><th>ref m.wave (NFP)</th></tr>
<tr><td>Test 01</td><td>1.2</td><td>0.3</td><td>0.8</td><td>0.8</td><td>0.3</td><td>0.3</td><td>0.3</td><td></td></tr>
<tr><td>Test 02</td><td>0.8</td><td>0.3</td><td>0.8</td><td>0.8</td><td>0.4</td><td>0.4</td><td>0.2</td><td>0.0</td></tr>
<tr><td>Test 03</td><td>1.1</td><td>0.3</td><td>1.0</td><td>1.0</td><td>0.7</td><td>0.5</td><td>0.3</td><td>0.2</td></tr>
<tr><td>Test 04</td><td>1.2</td><td>0.5</td><td colspan="2">0.9</td><td colspan="2">0.8</td><td>0.5</td><td>0.2</td></tr>
<tr><td>Test 05</td><td>0.9</td><td>0.5</td><td colspan="2">0.9</td><td colspan="2">0.6</td><td>0.3</td><td></td></tr>
<tr><td>Test 06</td><td>1.1</td><td>0.6</td><td>0.9</td><td>0.6</td><td>0.5</td><td>0.3</td><td>0.4</td><td>0.2</td></tr>
<tr><td>Test 07</td><td>1.1</td><td>0.1</td><td>0.7</td><td>0.6</td><td>0.4</td><td>0.1</td><td>0.2</td><td>0.0</td></tr>
<tr><td>Test 08</td><td>0.9</td><td>0.0</td><td>0.8</td><td>0.8</td><td>0.6</td><td>0.2</td><td>0.3</td><td>0.1</td></tr>
<tr><td>Test 09</td><td>1.2</td><td>0.4</td><td>0.8</td><td>0.9</td><td>0.4</td><td>0.3</td><td>0.2</td><td>0.1</td></tr>
<tr><td>Test 10</td><td>1.2</td><td>0.6</td><td colspan="2">1.1</td><td colspan="2">0.6</td><td>0.3</td><td></td></tr>
<tr><td>Test 11</td><td>1.1</td><td>0.2</td><td colspan="2">1.2</td><td colspan="2"></td><td>0.3</td><td>0.1</td></tr>
<tr><td>Test 12</td><td>0.8</td><td>0.2</td><td colspan="2">1.1</td><td colspan="2">0.2</td><td>0.5</td><td>0.2</td></tr>
<tr><td>Test 13</td><td>1.2</td><td>-0.1</td><td colspan="2">1.1</td><td colspan="2"></td><td>0.4</td><td>0.2</td></tr>
<tr><td>Test 14</td><td>1.2</td><td></td><td colspan="2">1.0</td><td colspan="2">0.2</td><td>0.4</td><td>0.2</td></tr>
<tr><td>Test 15</td><td>1.1</td><td>0.7</td><td>1.0</td><td>1.0</td><td>0.7</td><td>0.6</td><td>0.5</td><td>0.2</td></tr>
<tr><td>Test 16</td><td>1.4</td><td>0.6</td><td colspan="2">1.2</td><td colspan="2">0.6</td><td></td><td></td></tr>
<tr><td>Test 17</td><td>1.2</td><td>0.4</td><td colspan="2">0.8</td><td colspan="2">0.3</td><td></td><td></td></tr>
<tr><td>Test 18</td><td>1.1</td><td>0.4</td><td colspan="2">0.9</td><td colspan="2">0.3</td><td>0.5</td><td>0.2</td></tr>
<tr><td>Test 19</td><td>1.4</td><td>0.7</td><td colspan="2">0.8</td><td colspan="2">0.5</td><td>0.3</td><td>0.1</td></tr>
<tr><td>Test 20</td><td>1.3</td><td>0.6</td><td colspan="2">0.7</td><td colspan="2">0.2</td><td>0.3</td><td>0.1</td></tr>
<tr><td>Test 21</td><td>1.3</td><td>0.8</td><td colspan="2">1.1</td><td colspan="2">0.6</td><td>0.5</td><td>0.2</td></tr>
<tr><td>Test 22</td><td>1.0</td><td>0.8</td><td colspan="2">1.0</td><td colspan="2">0.3</td><td>0.0</td><td>0.0</td></tr>
</table>

Appendix 5, page 1: Microwave oven heating and total test durations , maximum and average temperatures measured during testing

	Sample	Series 1 (3200W)				Series 2 (1600W, 1st drying period long)				Series 3 (ASTM D 4643-08)			
		Heating	**Total**	**Max temp.**	**Avg temp.**	**Heating**	**Total**	**Max temp.**	**Avg temp.**	**Heating**	**Total**	**Max temp.**	**Avg temp.**
	number	[min]	[min]	[°C]	[°C]	[min]	[min]	[°C]	[°C]	[min]	[min]	[°C]	[°C]
0/1	**1**	7	10.4	134.9	111.2	18	21.4	111.7	99.3	10	13.4	111.7	94.4
	2	8	10.9	166.0	136.7	17	20.4	107.1	97.7	13	15.9	137.5	120.8
	3	10	12.9	171.7	122.8	16	18.9	153.3	105.8	10	13.4	122.5	93.5
	4	11	13.9	144.7	114.6	16	19.8	131.9	91.6	12	15.4	95.5	71.2
	5	9	12.4	168.8	157.1	11	13.9	129.9	102.1	10	13.8	111.2	87.6
	6	9	12.8	257.9	163.5	12	14.9	131.4	101.2	11	14.4	106.1	93.7
	7	8	10.9	149.8	106.0	13	15.9	112.1	99.5	11	14.4	79.7	64.6
	8	8	11.4	90.4	70.7	12	14.9	106.5	84.3	11	14.4	90.4	70.7
	9	6	8.9	112.3	94.4	13	15.5	106.7	90.4	12	15.4	84.7	68.8
	10	7	10.4	119.2	99.7	12	14.9	121.6	95.2	11	14.4	83.8	71.9
0/6	**1**	7	10.4	191.7	138.8	20	23.4	187.3	148.9	10	12.9	150.0	123.2
	2	8	10.9	162.7	130.4	17	20.4	159.7	130.6	13	16.4	149.1	124.4
	3	10	12.9	240.3	182.8	16	18.9	196.0	117.6	10	13.4	109.6	86.2
	4	11	13.9	216.3	167.1	16	18.9	203.1	126.0	12	15.4	164.2	107.8
	5	9	11.5	238.3	177.9	14	16.9	190.9	133.7	10	13.4	154.4	110.6
	6	9	11.5	284.6	173.3	12	14.5	167.2	126.6	11	14.4	132.4	92.4
	7	8	10.9	237.7	157.1	13	16.8	182.9	146.1	11	13.9	153.9	110.1
	8	8	11.4	231.2	167.8	12	14.9	163.8	117.0	11	14.4	151.1	102.5
	9	6	8.9	232.7	144.9	13	16.4	162.6	123.8	12	15.8	127.3	93.5
	10	7	10.4	215.1	154.2	11	13.5	160.8	125.2	11	14.4	153.4	114.8

Appendix 5, page 2: Microwave oven heating and total test durations, maximum and average temperatures measured during testing

	Sample	Series 1 (3200W)				Series 2 (1600W, 1st drying period long)				Series 3 (ASTM D 4643-08)			
		Heating	Total	Max temp.	Avg temp.	Heating	Total	Max temp.	Avg temp.	Heating	Total	Max temp.	Avg temp.
	number	[min]	[min]	[°C]	[°C]	[min]	[min]	[°C]	[°C]	[min]	[min]	[°C]	[°C]
4/8	**1**	6	9.4	213.8	131.2	13	16.4	267.7	232.3	9	12.4	164.2	115.0
	2	8	11.8	291.7	131.1	14	17.4	159.3	105.3	11	14.4	128.9	110.1
	3	8	11.4	211.7	140.1	14	16.9	186.5	138.7	8	11.4	119.4	76.8
	4	8	11.4	223.8	181.1	11	14.4	161.0	98.6	11	14.8	150.3	88.9
	5	9	12.8	240.4	212.7	11	13.9	184.2	124.1	9	12.8	145.6	110.3
	6	8	11.4	224.3	137.7	10	12.9	140.8	123.8	11	14.4	151.9	116.2
	7	7	10.4	192.7	132.3	10	12.9	214.5	144.6	8	10.9	133.6	93.7
	8	8	10.9	303.5	183.2	8	10.9	139.9	94.9	9	12.4	129.1	94.3
	9	5	7.9	271.8	187.3	8	10.5	132.8	97.2	10	13.8	128.7	87.6
	10	7	10.4	305.2	192.8	8	10.5	146.1	99.4	9	12.4	169.9	111.9
8/16	**1**	5	7.9	421.7	292.1	13	15.9	271.1	213.8	9	12.4	313.2	171.6
	2	7	10.4	446.0	273.5	13	15.9	298.4	196.8	12	15.8	407.1	263.4
	3	7	9.9	430.0	312.2	13	15.5	552.2	292.6	8	11.4	290.2	135.3
	4	8	11.4	469.0	316.4	11	13.9	405.4	286.3	11	14.8	285.2	229.7
	5	8	11.4	441.9	317.2	11	13.9	359.3	274.7	9	12.8	321.9	245.3
	6	8	11.4	441.7	339.4	11	14.4	286.6	220.8	11	14.4	311.6	239.4
	7	8	11.8	543.0	395.3	10	12.9	251.4	168.7	8	10.9	304.8	180.3
	8	Testing broken off				9	11.9	305.6	197.4	9	12.4	269.7	198.6
	9					8	10.5	296.7	206.4	10	13.8	319.7	194.6
	10					8	10.5	256.6	189.1	9	12.4	237.9	199.6