Water Sorption of Hardened Cement Pastes

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<http://dx.doi.org/10.5772/intechopen.76378>

Abstract

Hardened cement paste (hcp), binding the components in cementitious composites, usually controls most strength, transport, and durability properties of these materials. Water sorption in hcps can cause durability problems such as sulfate and chloride ingress, frost deterioration, and esthetic problems. Replacement of air by water in the pores can also increase the thermal conductivity of the material and affect the energy efficiency. Capillary suction test as a simple method for characterization of the material resistance to water sorption is described in this chapter. Different factors affecting water sorption of hcps such as changing water to cement ratio (w/c), using pozzolanic materials, and internal hydrophobation are also discussed. Furthermore, resistance number, capillary number, and pore protection factor as different criterions for characterizing the moisture transport in cement-based materials are described. Since cement-based materials modified for reducing water sorption have different behavior in capillary suction test compared to ordinary materials, the abovementioned criteria may become inapplicable for characterizing these materials. Thus, “effective moisture transport (EMT)” factor is introduced here which can be a more comparative measure for modified cementitious materials with denser or internally hydrophobed pore structure.

Keywords: cement paste, hcp, water sorption, pozzolanic materials, capillary suction, resistance number, hydrophobic agent, silane, silica

1. Introduction

Water sorption of hardened cement pastes (hcps) is one of the key factors that can affect particular properties of cement-based materials. Chloride and sulfate ingress as well as frost deterioration and esthetic appearance are some of durability problems that are caused by water transport in these materials. Thermal properties are also affected by changes in water

sorption of cementitious composites. Reducing moisture content can, for example, lower thermal conductivity and reduce heat exchange through the materials in the building envelope.

Water is mainly transported through the capillary pores in hcps. Since increasing water to cement ratio (w/c) increases capillary porosity, the first solution to reduce water permeability is usually reducing w/c [1]. Using pozzolanic materials such as silica fume, fly ash, and slag can also result in a denser pore structure and average pore size, which results in more resistance to water sorption. The other method to modify water permeability in cement-based materials is using hydrophobic agents. Both the material surface and the internal structure can be made water-repellant by using these agents [2–5]. While UV light of the sun can affect water repellence of the surface-treated material, internal hydrophobation does not have this drawback.

In this chapter, water sorption of hcps as the important factor in permeability of cement-based materials is described. Moreover, the resistance number and the capillary number [6], which are calculated based on capillary suction test, will be described here. These numbers are used for the characterization of the material resistance to water sorption. However, this method is not suitable for modified structure of hcps that have high resistance to water sorption. Thus, an alternative parameter “effective moisture transport (*EMT*)” factor is proposed in this chapter instead of the resistance number according to the experimental results.

2. Capillary suction test

Smeplass and Skjølsvold [6] improved the test for capillary suction by including information about the pore structure of hardened concrete. Martys and Ferraris [7] have also studied capillary transport in mortars and concrete. In order to perform the capillary suction test, the specimens are usually placed in a ventilated incubator at 105°C until reaching constant weight. Lower temperatures (e.g., at 50°C in an oven or at 20°C in a desiccator) with longer drying interval may also be chosen to reduce the effect of drying on pore structure and composition of the material components. After drying, the specimens are placed on a grating 1–2 mm below the water surface. The test is usually performed in the following order:

1. drying to a constant weight,
2. capillary suction for 4–5 days,
3. submersion in water for 3–4 days at 1 atm,
4. submersion in water for 1–3 day at 50–80 atm,
5. recording the specimen volume,
6. drying the specimen at 105°C to reach a constant weight.

The duration of each step may vary depending on the water resistance of the sample as well as its height. For ordinary materials, the standard test procedure in each country may be used; however, for modified materials, it may be necessary to adjust the test parameters such as duration and water pressure.

3. Specimen preparation for capillary suction test

In case of concrete samples casted in the laboratory, 20-mm slices from cylinders cured for 28 days are usually cut for the test. To avoid material spalling from edges due to cutting, epoxy or other hard-wearing coating can be applied on the cylinders. **Figure 1** illustrates an example of sample preparation from concrete cylinders.

A similar procedure may be used for preparing hcp samples. The size of these specimens is usually smaller than concrete samples. The mixing procedure for hcps may also be different from ordinary concrete. Using a high-speed kitchen blender to facilitate proper mixing

Figure 1. Cutting specimens from concrete cylinders for capillary suction test [4].

of water and cement is popular in this process. The following order may be carried out for mixing: a 1-min medium rate mixing; cleaning the mixer wall with a flexible plastic spatula; a 4-min fast rate mixing; cleaning the mixer wall with a spatula and finally a 1-min slow rate mixing. Moreover, the admixtures (such as hydrophobic agents, plasticizer air-detraining admixture) may be mixed with water for 1 min, before adding to the cement to assure proper dispersion in the cement paste. The specimens with high w/c that have risk of separation may be cured on a slow-rate rolling machine on the first day.

4. Parameters affecting water sorption of hcps

Changes in the pore structure by altering w/c or adding pozzolanic materials as well as incorporating hydrophobic agents are the main parameters affecting the water sorption of hcps. In this part, the effect of each parameter will be illustrated by presenting the results from experimental

Mix type	w/c	Alkyl alkoxysilane (% m _c)	Rapeseed oil (% m _c)	Silica fume (% m _c)	Plasticizer (% m _c)	Air-detraining admixture (g/l)
Ref	0.58	0	0	0	0.6	8
	0.44	0	0	0	0.6	8
	0.36	0	0	0	0.6	8
Si	0.58	1	0	0	0.6	8
	0.44	1	0	0	0.6	8
	0.36	1	0	0	0.6	8
Oil	0.58	0	1	0	0.6	8
	0.44	0	1	0	0.6	8
	0.36	0	1	0	0.6	8
SF	w/b = 0.48	0.58	0	20	0.72	8
	w/b = 0.40	0.44	0	10	0.66	8
	w/b = 0.37	0.44	0	20	0.72	8
	w/b = 0.34	0.44	0	30	0.78 (SP)	8
	w/b = 0.30	0.36	0	20	1.44 (SP)	8

Table 1. Mix proportions for reference (Ref) samples and the sample containing rapeseed oil (Oil), alkyl alkoxysilane (Si), and silica fume (SF).

investigations [5, 8]. **Table 1** presents mix proportions for four mix series of hcps. The effect of changes in w/c is investigated by considering three different ratios of 0.58, 0.44, and 0.36 in each series.

Portland cement (CEM I 45.5 R) with a specific area of 550 (m²/kg) and a specific density of 3120 (kg/m³) were used. The series include reference (Ref) which is plain hcp, two series containing hydrophobic agents, and one series containing silica fume (SF) as pozzolanic material.

One percent of cement mass (mc) of a silane-based product (100% alkyl alkoxysilane) which has a small molecular size of 5–10 Å was used in the “Si” series. This material is developed for surface hydrophobation but it was used here as an admixture to study its effect for internal hydrophobation. In addition, food quality rapeseed oil with 8% saturated, 62% monounsaturated, and 30% polyunsaturated fatty acids was used as the other hydrophobic admixture in the “Oil” series with a dosage of 1% mc. The oil was selected as an environmental friendly substitute for existing chemical hydrophobic agents [9, 10]. Today, there are different hydrophobic agents in the market as concrete admixture where the producer claims effective hydrophobicity and no negative effect on mechanical properties; the selected hydrophobic agents here are to show how these agents may affect the water sorption of hcps.

The “SF” series contain densified silica fume with a specific density of 2200 (kg/m³) and a dosage of 20% mc. This high dosage was considered to see the effect of finer pore structure in water suction of hcps.

In order to reduce undesirable macro-air pores, an air-detraining admixture based on modified polysiloxanes with 0.5% dry matter was used in all of the mixes. In addition, a plasticizer based on ligno-sulfonates with 40% dry matter and a super plasticizer (SP) based on modified acrylic polymers with 30% dry matter were used in the cement paste mixes. The composition of the mixes is listed in **Table 1**. The water to binder ratio (w/b) is also given for the SF series in this table since pozzolanic materials work as a part of binder in cementitious composites.

The samples were cured in water for 12 weeks, and then dried in ventilated oven for 2 weeks at 50°C. Parameters obtained from capillary suction test are given in **Table 2**. Capillary suction porosity (ϵ_{csuc}), capillary submersion porosity (ϵ_{csub}), and pressure saturated porosity (ϵ_{ps}) are calculated from the mass gain after capillary suction, submersion in water at 1 (atm) and submersion in water at 50 (atm), respectively. Furthermore, **Figures 2–4** show the pore distribution of different mixes with the same w/c. Note that this test is not an accurate test for porosimetry and is intended for characterizing material behavior under water sorption. The results show that the total capillary porosities ($\epsilon_{\text{csuc}} + \epsilon_{\text{csub}}$) are decreased for the Oil samples due to reduction in water absorption by using hydrophobic agents. Unlike impregnating agent emulsion that was used by Haugan [4], alkyl alkoxysilane had a minor effect on internal hydrophobic treatment for all the three selected mixes with different w/c, showing that not all the hydrophobic agents developed for surface treatment are also effective in reducing water sorption when used as admixture.

Furthermore, using silica fume has decreased ϵ_{csuc} and increased ϵ_{csub} and it is more obvious in lower w/c. In fact, there is a minor reduction in the total capillary porosity of SF samples, but the reason and amount of this reduction is different from the Oil samples. Moreover, considering Ref and SF samples with w/b in the same range, we can see that although the total porosity has slightly increased for SF samples, ϵ_{csuc} has decreased, indicating more resistance to water transport in SF samples due to reduction in pore size and connectivity between the pores.

The oil shows the best effect on reducing absorption, with a large amount of pore space that can only be accessed by high water pressure. The performance of oil is probably due to some water repellency effect, whereas the performance of silica fume is due to a reduction in pore size and connectivity between the pores. In fact, changes in the pore structure by altering w/c have only changed the total porosity of the material and did not have a significant effect on reducing the capillary suction of hcps. Adding silica fume had a minor effect on reducing the total capillary water absorption ($\epsilon_{\text{csuc}} + \epsilon_{\text{csub}}$) but decreased the capillary suction by creating a denser pore structure. However, using a low amount of proper hydrophobic agent as admixture can reduce the water sorption of hcps significantly.

In addition to the results shown in **Table 2**, the degree of hydration was calculated from w/c and ϵ_{tot} shown in this table to calculate the dry sample density (ρ_d) according to Power's model [11] which agreed very well with measured ρ_d (mainly less than 2% difference). Thus, these hcps behave as they should in terms of Powers model.

“Pore protection factor” (PF) is a criterion for assessing frost resistance of concrete in Finnish Standard SFS 4475 [12]. It is defined as the air content as a percentage of the total porosity ($\text{PF} = \epsilon_{\text{air}}/\epsilon_{\text{tot}}$). It is worth noting that ϵ_{ps} is usually considered as ϵ_{air} for normal concrete, but the abovementioned hcps contain a low amount of air pores as judged from **Table 2** for the

Mix type	w/c	ϵ_{enc} (vol%)	ϵ_{emb} (vol%)	ϵ_{ps} (vol%)	ϵ_{tot} (vol%)	ρ_s (kg/m ³)	ρ_d (kg/m ³)
Ref	0.58	49.2 ± 0.5	1.1 ± 0.6	0.4 ± 0.1	50.7 ± 0.2	2696 ± 2	1329 ± 4
	0.44	42.3 ± 0.1	0.4 ± 0.0	0.4 ± 0.0	43.1 ± 0.1	2712 ± 3	1543 ± 3
	0.36	35.8 ± 1.4	1.5 ± 1.4	0.6 ± 0.1	38.0 ± 1.4	2737 ± 10	1698 ± 6
Silane	0.58	46.6 ± 0.7	2.7 ± 0.5	1.7 ± 0.3	51.1 ± 0.1	2686 ± 2	1315 ± 3
	0.44	39.9 ± 0.4	1.7 ± 0.6	1.3 ± 0.5	43.0 ± 0.3	2675 ± 7	1526 ± 14
	0.36	33.4 ± 3.3	4.0 ± 3.2	1.0 ± 0.1	38.4 ± 0.1	2724 ± 3	1677 ± 2
Oil	0.58	29.8 ± 1.9	12.0 ± 1.4	7.4 ± 0.8	49.2 ± 0.1	2631 ± 5	1335 ± 2
	0.44	22.7 ± 1.8	6.5 ± 1.5	12.2 ± 1.0	41.1 ± 0.4	2642 ± 6	1548 ± 14
	0.36	19.6 ± 0.6	3.8 ± 1.6	13.3 ± 1.1	36.7 ± 0.1	2679 ± 2	1695 ± 4
Silica fume	0.58	43.1 ± 0.8	2.3 ± 0.9	2.0 ± 0.3	47.4 ± 0.1	2687 ± 2	1413 ± 2
	0.44	37.4 ± 0.5	1.9 ± 0.2	2.2 ± 0.4	41.5 ± 0.1	2704 ± 11	1582 ± 10
	0.44	33.1 ± 2.4	4.8 ± 2.2	2.9 ± 0.1	40.8 ± 0.1	2716 ± 6	1607 ± 1
	0.44	26.0 ± 2.0	10.7 ± 1.7	2.8 ± 0.5	39.5 ± 0.2	2699 ± 4	1632 ± 4
	0.36	25.2 ± 0.8	7.9 ± 0.7	2.5 ± 0.3	35.6 ± 1.2	2719 ± 31	1751 ± 12

Table 2. Capillary suction porosity (ϵ_{enc}), capillary submersion porosity (ϵ_{emb}), pressure saturated porosity (ϵ_{ps}), total porosity (ϵ_{tot}), average density of solids (ρ_s) and dry sample density (ρ_d), derived from capillary suction test.

Figure 2. Pore distribution of different mixes with the w/c of 0.36 for reference (Ref) sample and the samples containing 1% rapeseed oil (Oil), 1% alkyl alkoxysilane (Si), and 20% silica fume (SF).

Figure 3. Pore distribution of different mixes with the w/c of 0.44 for reference (Ref) sample and the samples containing 1% rapeseed oil (Oil), 1% alkyl alkoxysilane (Si), and 20% silica fume (SF).

reference materials. On the other hand, in case of internal hydrophobation of these samples, a considerable amount of the capillary pores are not filled after 3 days of submersion in water at 1 atm but they fill at 50 atm. The apparent air voids may thus be a part of ϵ_{ps} . **Figure 5** shows PF values for different mixes by considering ϵ_{ps} as ϵ_{air} . According to this figure, it is concluded that although silica fume gives a denser pore structure, it has a minor effect on blocking the pores from the suction of water at atmospheric pressure. On the contrary, oil has been effective in increasing the PF value, indicating water repellency effect in the pores. This effect has been increased by a reduction in w/c where the overall pore size becomes smaller.

Eq. (1), La Places or Washburn's equation, shows the pressure that forces water with surface tension between air and water (σ) into a pore of radius (r). Since the contact angle for the

Figure 4. Pore distribution of different mixes with the w/c of 0.58 for reference (Ref) samples and the samples containing 1% rapeseed oil (Oil), 1% alkyl alkoxysilane (Si), and 20% silica fume (SF).

Figure 5. Pore protection factor (PF) for reference (Ref) sample and the samples containing rapeseed oil (Oil), alkyl alkoxysilane (Si), and silica fume (SF).

hydrophobed surface is more than 90° , the pressure sign will be positive, thus the smaller the pore radius, the larger the repellency effect if these small pores are hydrophobed. The pore structure of hcp is more complicated than a capillary tube with connections between the pores. In addition, the hydrophobic agents may not cover all the pore surface areas. One may generally assume that the pore structure of hcps with lower w/c is finer than the higher w/c, simply by comparing the volumetric fraction of gel pores. Consequently, it is expected that water-repellant admixtures will be more effective in lower w/c if these smaller pores are the main part of the pore system that is impregnated. This effect is observed in the current example as an increase in ε_{ps} by using hydrophobic agents (**Figure 6**).

$$P = \frac{(-2\sigma \cos\theta)}{r} \quad (1)$$

Figure 6. Hydrophobic and non-hydrophobic surfaces [13].

Note that the behavior of hcp to water sorption may have some differences with concrete specimens. Hardened cement paste gets some micro-cracks during drying period. Visual observations of micro-cracks on these pastes indicate that they cause faster water suction when placed on the water surface. These micro-cracks will, however, close after water absorption and are not expected to highly change the total porosity, but they may open some of the pores that were not accessible to capillary water and reduce pressure-saturated porosity. Since the effect of cracking can be more in higher w/c due to a higher amount of capillary water, the PF value may be less for higher w/c compared to lower w/c for Oil samples. On the other hand, the interfacial transition zone (ITZ) between the cement paste and aggregates in concrete that is the weak part of the concrete matrix can be the reason for different behavior of hydrophobic agents in hcp and concrete.

In addition, pore blocking by oil droplets and denser pore structure are the other possible reasons that have been mentioned by [3] for less PF values in some concrete samples. The effect of denser pore structure can be seen in **Figure 5** by comparing the PF value for Ref samples with w/c = 0.36 and SF samples with w/c = 0.44 which have a w/b of 0.37. The PF values for SF samples are higher than Ref samples due to denser and more discontinuous pore structure, but this effect is not comparable to the water repellency effect of rapeseed oil.

5. Effective moisture transport factor

In order to measure the resistance to capillary suction, Smeplass and Skjølsvold [6] have suggested calculating the time, t_{cap} , and the corresponding absorption value, Q_{cap} , that the water front reaches the top surface of the specimen with the height “h” (see **Figure 7**). The mass storage is usually registered in the following order during capillary suction test:

- 10 and 30 min,
- 1, 2, 3, 4, and 6 h,
- 1, 2, 3, 4 and alternatively 5 days.

Figure 7. Regression analysis for calculating the resistance number (m) and the capillary number (k).

A regression analysis is then carried out based on the data points from 10 min to 6 h for the first/left linear part, and the rest of the points (from 1 to 4 days or alternatively 5 days) for the second/right linear part. Then, the resistance number, m , and the capillary number, k , will be calculated using the following equations:

$$m = \frac{t_{cap}}{h^2} \quad (2)$$

$$k = \frac{Q_{cap}}{\sqrt{t_{cap}}} \quad (3)$$

Figure 8. Water absorption versus the square root of time of different mixes with the w/c ratio of 0.36 for reference (Ref) samples and the sample containing 1% rapeseed oil (Oil), 1% alkyl alkoxysilane (Si), and 20% silica fume (SF) ($w/b = 0.30$).

The resistance number and the capillary number reflect the fineness of the pore system. In cementitious composites, the capillary number indicates the amount of the binder in the material by neglecting water sorption of the other composite components. However, these values cannot be properly calculated to give a good reflection of the material behavior under capillary suction in the current SF and Oil samples. The reason is high resistance of these specimens to water suction which does not give a clear nick point for calculating t_{cap} and Q_{cap} . Thus, another method for characterization of these samples is desired which can give a proper comparison between ordinary materials and the modified ones. *EMT* factor will be defined in this part for this purpose.

Figure 8 presents the water absorption versus the square root of time for specimens with $w/c=0.36$. As seen in the figure, although silica fume does not have a high effect on PF, it reduces capillary suction compared to reference and silane samples. The reason can be described as the finer pore structure as well as less connection between the pores (less percolated), but since it does not repel water or block the pores, the pores will more easily be filled with water than hydrophobized samples in case of submersion. Moreover, the Ref samples have a higher absorption than the other samples since they have less resistance to water transport in the pores. On the other hand, although the rate of mass transport (the slope of the lines in **Figure 8**) in SF and Oil samples is similar in this case, but the absorption in SF samples is larger than Oil samples. This is probably due to the initial moisture content of SF samples. In other words, SF and Oil samples have shown similar resistance to water suction according to **Figure 8**, but since the SF samples had a higher moisture content after drying at 50°C, the moisture content is higher for SF samples after the water suction test. Since the samples have been dried in the oven for 2 weeks at 50°C, it can be judged that the moisture content after this drying period is not very active in water transport during capillary suction test. Therefore, by considering the initial moisture as mass content with low mobility, we can define the mobile capillary suction porosity (ϵ_{mcsuc}) as the weight gain after capillary suction excluding initial moisture (**Table 2**). This can be a more realistic estimation of the part of the pore structure which is involved in capillary suction compared to the value obtained after severe drying at 105°C which highly affects the pore structure of the material as well.

The slope of the line obtained from the first 6 h of suction curve, K'' , can be an indication of the rate of mass transport in the material. Furthermore, the final mass storage after water suction test could be different for the samples with the same K'' due to different gradients in the curves after the first 6 h. In other words, ϵ_{mcsuc} can be different for the samples with the same K'' due to different pore structure or pore chemistry. Thus, both ϵ_{mcsuc} and K'' are indications of moisture transport in the material in the abovementioned capillary test. A general experience with capillary suction testing of cement-based materials is that the capillary nick points become less clear at reducing w/b , when adding pozzolana, increasing the initial moisture content and in hydrophobed samples. In such cases, the resistance number and the capillary number are not useful, and therefore a different parameter is proposed here: “effective moisture transport (*EMT*)” factor. *EMT* can be defined as a criterion for the effective mass transport in the material where we have problems of defining a nick point:

$$EMT = \sqrt{\epsilon_{mcsuc} \times K''} \quad (4)$$

Figure 9. Effective moisture transport factor (*EMT*) for reference (Ref) samples and the sample containing 1% rapeseed oil (Oil), 1% alkyl alkoxysilane (Si), and silica fume (SF).

Figure 10. Effective moisture transport factor (*EMT*) for reference (Ref) samples and the sample containing 1% rapeseed oil (Oil) and 10, 20, and 30% silica fume(SF) as a function of water to binder (*w/b*) ratio.

where ϵ_{msuc} is the mobile capillary suction porosity and K'' is the slope of the line obtained from the first 6 h of suction curve. Both ϵ_{msuc} and K'' depend on the fineness, connectivity, chemistry, and volume of the mobile capillary suction pores in the material but from different perspectives; thus, a square root of multiplication of these two values can be a criterion for effective mass transport in the material.

Figure 9 shows *EMT* for different *w/c* ratios of the mixes. The effect of fine pore structure is shown for both adding the same amount of silica fume to different *w/c* ratios and increasing the dosage of silica fume for a constant *w/c* ratio. All correlation coefficients from the regression

analysis of the slopes were more than 0.93. Since the slopes of the lines for both SF and Oil have been similar during the first 6 h and the rest of the capillary suction test (**Figure 8**), a similar *EMT* value has been obtained for these samples. Furthermore, the difference between the resistance of SF to water suction compared to Si and Ref samples is clearer in **Figure 9**. Moreover, the figure shows that using 1% rapeseed oil has a reduced *EMT* of the hcp with $w/c = 0.58$ to a level even lower than the reference material with $w/c = 0.36$. Using 30% SF was as effective as using 1% rapeseed oil in samples with $w/c = 0.44$. It is worth noting that adding silica fume is more effective in resistance to mass transport than reducing w/c ratio. This is shown in **Figure 10** in which the effect of adding 10, 20, and 30% silica fume in reducing *EMT* is compared to the Ref and Oil samples as a function of water to binder ratio (w/b). However, 1% oil is found to be more effective than using SF.

6. Conclusions

Factors affecting the water sorption of hcps such as w/c , pozzolanic materials, and internal hydrophobation were described in this chapter. The hcps with lower w/c have a less total porosity which results in a less water sorption. The amount of pores filled with water under capillary suction (ϵ_{suc}) was near to total porosity (ϵ_{tot}) for plain hcps (Ref samples). In addition, silica fume as pozzolanic material increases resistance to water transport in hcps due to a reduction in the pore size and the connectivity between the pores, but it is not effective in reducing the total water suction of hcps.

A minor effect on water sorption was observed using alkyl alkoxysilane showing that this agent which is developed for surface treatment is not suitable for internal hydrophobation. However, rapeseed oil as a hydrophobic agent resulted in an obvious reduction in the water suction of hcps. Pore blocking by oil droplets and denser pore structure can be the other possible reasons for less PF values in some samples. However, the effect of a denser pore structure is not comparable to the water repellency effect of the oil. The behavior of hcps under water suction can be different from cement-based composites due to the effect of ITZ in the composite materials and the formation of micro-cracks in hcps during drying.

Due to the lack of clear capillary nick points, for the determination of resistance number and capillary number, an alternative parameter “effective moisture transport (*EMT*)” factor was proposed instead in this chapter. The *EMT* factor can be a more comparative measure for denser or hydrophobed samples, especially when the samples are dried at lower temperatures to reduce the effect of drying on pore structure and composition of the material components.

Acknowledgements

The author gratefully appreciates the Norwegian University of Science and Technology (NTNU) for project funding, professor emeritus Per Jostein Hovde, professor Stefan Jacobsen, and adjunct professor Roar Myrdal at NTNU for their advices as well as the help given by engineer Ove Edvard Loraas in the NTNU laboratory.

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