Report

Acid Activation of Natural Rocks

A Case Study with Norwegian Anorthosite and Phosphoric Acid

Author(s)
Tobias Danner
Harald Justnes

Anorthosite from Gudvangen, Norway (Foto: geocaching.com/Sveinivar)
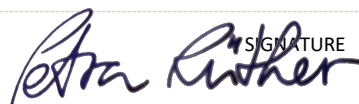
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ABSTRACT

Norway is rich in natural resources, especially different types of rocks that could potentially serve as raw material for alternative binder concepts like alkali activated binders or magnesium phosphate and magnesium hydroxy carbonate cements. Another less studied concept, as discussed in this paper, is acid activation of rocks to produce alternative binders. Although these systems cannot compete yet with ordinary Portland cement, they might be applicable for certain specialised areas and thus help to reduce cumulated CO_2 emissions by decreasing the total amount of cement used. Alternative binder systems based on Norwegian rocks as raw material show potential for further research and potential implementation of new specialised products for the domestic market. To demonstrate the potential of acid activation, first results of a case study on anorthosite activated by phosphoric acid are presented. Results show that it was possible to produce a binder with 28-day compressive strength of about 20 MPa, by mixing warm phosphoric acid with finely ground Norwegian anorthosite. The paper presents preliminary results and further optimization, and research is necessary to produce higher strength and understand the hardening mechanisms of the acid activated anorthosite binder.

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Table of contents

1 Introduction

Cement is the largest manufactured product on earth with about 4.1 billion tonnes produced in 2017 [1], and it is well known that the production of ordinary Portland cement results in high emissions of CO_2. On a global average, about 842 kg CO_2 are produced per ton of cement [2] and is estimated that cement production contributes between 5-8 % of total anthropogenic CO_2 emissions. Therefore, the cement industry is working intensively with different options to reduce the CO_2 emissions associated with the production of cement and the final end user products like concrete. One of the main and most promising strategies is to reduce the clinker content in binders by increasing the amount of low CO_2 supplementary cementitious materials (SCMs) [3]. Currently, Norcem AS replaces between 15 and 20 % of the Portland clinker by fly ash in about 80 % of the cement produced for the domestic market. For the future, it is aimed to reduce the clinker content further by increasing the amount of SCMs to up to 35 % as in the recently developed FUTURECEM concept by Aalborg Portland [4].

Another concept is to reduce the total amount of ordinary Portland cements in use by producing alternative low carbon binders and cement clinkers. The most promising alternatives today, with potential for large scale implementation include a) reactive belite-rich Portland cement (RBPC) clinkers, b) belite-ye'elemite- ferrite (BYF) clinkers, c) carbonatable calcium silicate clinkers (CCSC) and d) magnesium oxide derived from magnesium silicates (MOMS) [5]. Another well-known concept is that of alkali activated binders or "geopolymers" [6]. Alkali activated binders are based on the reaction of an aluminosilicate precursor (e.g. fly ash, slags) with an alkaline solution to produce a hardened product of hydrous alkali-aluminosilicates and/or alkali-alkali earth aluminosilicate phases [6].

Norway is rich in natural rock and mineral resources and besides other applications, the potential of different types of rocks as partial replacement for cement is discussed and tested on selected materials frequently. Although, often indicating a favourable chemical composition, Norwegian rocks are mainly composed of highly crystalline minerals with low solubility in common cementitious systems based on Portland cement. Hence, most materials, finely ground without further treatment will only act as a pure filler in mortar or concrete. However, there might be potential for certain types of resources to be employed for concepts like magnesium-based binders or alkali activated binders. Large deposits of e.g. magnesite ($MgCO_3$), brucite ($Mg(OH)_2$), olivine (($Mg, Fe)_2SiO_4$), and serpentine (($Mg,Fe)_3Si_2O_5(OH)_4$) can be found that could be available for the production of MgO for e.g. magnesium phosphate and magnesium hydroxy carbonate cements [7]. On the other hand, Norwegian bedrock contains high amounts of aluminosilicate rocks like feldspars and there are large deposits of almost pure feldspar rocks like, norite and anorthosite. Due to their chemical composition (rich in Al and Si), these rocks might have the potential to be used as precursors for alkali activated binders when finely ground. Recently researchers from the University of Stavanger (Norway) started to evaluate the potential of Norwegian rocks for geopolymerization [8, 9].

Another approach by Justnes and Østnor investigated the possibility of producing pozzolanic materials from otherwise inert Norwegian rocks (olivine, serpentine and anorthosite) by chemical treatment with strong and weak acids [10, 11]. Reactive silica was obtained by e.g. dispersing finely ground olivine in water and hydrochloric acid. The synthesized silica showed a pozzolanicity similar to regular silica fume. Sequestration of CO_2 by olivine and serpentine producing $MgCO_3$ is also discussed in the report by Justnes [10]. Jahren and Lindbak reported about a new pozzolan consisting of a pure unstable amorphous SiO_2 phase that was produced as a by-product during the treatment of anorthite ($CaAl_2Si_2O_8$) with hydrochloric acid [12]. The pozzolan called "Sican" had a reactivity comparable to silica fume. As natural rocks can have a good

solubility in acids (depending on the mineralogical and chemical composition of the rock, the type of acid, and the treatment conditions, e.g. temperature and pressure), "acid activated binders" produced from natural rocks (in contrast to alkali activation) might introduce another new alternative concept to produce binders with low CO_2 emissions. At the same time, it may be possible to utilize and neutralize waste acids from other industries.

Recently a Canadian company announced the development of a CO_2 free white cement with high chemical and heat resistance, from white mountain anorthosite [13]. The product called "Anocrete" was made from white anorthosite from Greenland, phosphoric acid, water, aggregates and fibres and a compressive strength comparable to ordinary Portland cement (OPC) concrete was claimed [14]. Unfortunately, there is no scientific publication available on the topic yet, i.e. there is only little information available on the experimental procedure, parameters and results in comparison to cement pastes or mortar/concrete made with OPC.

Anorthosite is a common rock type occurring in many different localities of Norway. Actually, the two largest anorthosite occurrences in Western Europe can be found in Western Norway in the Sogn-Voss Province and the Rogaland Province [15]. The anorthosite occurrence in the Sogn-Voss and Rogaland province spreads over 700 and 500 km^2, respectively. Anorthosite is a rock type from the gabbro group that contains more than 90 % plagioclase (mainly anorthite). The residual crystalline fraction consists usually of pyroxenes, hornblende or micas (biotite, muscovite). Some anorthosite locations are known to be important ore deposits, containing ilmenite which can serve for the extraction of titanium. This makes these locations very attractive for mining and separation of the ore rich phases, resulting in huge amounts of mining tailings dumped without further application. It is estimated that about 4 million tonnes anorthosite mining waste is produced annually from ilmenite production [16].

The high Al_2O_3 content in anorthosite (~30 %) is attractive for several industries. Leaching of Al, Ca or Si by alkaline solutions or mineral acids like HCl or H_2SO_4 is a well-established process for various industrial applications of anorthosites [15]. Waste acids from other production lines might be used for this process (e.g. HCl waste from Borregaard or H2SO4 waste from Titania). Plagioclase is the name of a group of feldspar minerals that form solid solutions ranging from pure albite ($NaAlSi_3O_8$) to pure anorthite ($CaAl_2Si_2O_8$). The solubility of plagioclase in mineral acids is highly dependent on the anorthite content. While plagioclase with an anorthite content below 50 % has almost no solubility for Al_2O_3, almost 100% Al_2O_3 can be leached from plagioclase rocks with an anorthite content above 80% [17, 18]. The massifs from the Sogn-Voss Province contain high quantities of easily soluble anorthosite with an Al_2O_3 content around 30 %. Hence, anorthosite was considered as an alternative source of aluminium ore for the Norwegian aluminium industry, and the feasibility of replacing bauxite as a raw material for aluminium production was investigated [19, 20]. A special white altered variety of anorthosite is sometimes used for aesthetic reasons as aggregate in concrete elements in e.g. exterior walls or used for filler purposes [15].

Based on the Canadian "Anocrete" concept [13], it was attempted to reproduce some of the claimed results by investigating the possibility of producing a binder from Norwegian anorthosite rock and phosphoric acid similar to the binder described in. This article summarizes first laboratory small scale attempts on paste level to activate Norwegian anorthosite rock by phosphoric acid with the aim to form a new type of binder developing enough strength to be used in specific applications.

2 Materials

Two different samples of anorthosite rock from the Sogn-Voss Province were investigated. One fresh (unaltered) and one altered anorthosite [15]. Alteration of rocks refers to geological processes that cause changes in the chemical and mineralogical composition since the rocks original formation. The unaltered anorthosite (An-ua) was greyish in colour while the altered anorthosite (An-a) had a white colour. Both materials were received in coarse fractions with grain sizes up to 5 mm. The materials were milled down to a median particle size < 30 µm. Milling was done with an "auto-mortar" (Retsch RM 200) for 10 minutes at highest pressure. The particle size distribution of both materials can be seen in Figure 1. After milling, the unaltered anorthosite was slightly coarser. The chemical composition of both materials is given in Table 1. Both materials are very similar in chemical composition with about 50 % SiO_2, 30 % Al_2O_3 and 14 % CaO typically observed for anorthosite from the Sogn-Voss Province [21]. An approximate mineralogical composition determined with X-Ray diffraction is given in Table 2.

Both samples contain more than 90% feldspar minerals. The phase assemblage of both samples is similar. However, the feldspars in the unaltered rock sample contain a higher amount of anorthite content compared to the altered rock [21]. On the other hand, the altered rock contains somewhat more alteration products like micas, clays, zeolites (see minor phases in Table 2). The higher amount of alteration phases is reflected in the slightly higher weight loss (LOI) of the altered anorthosite sample due to loss of structural water upon heating. According to [21], the unaltered anorthosite has an anorthite content between 65-78 %, while the altered type has a lower anorthite content than the unaltered one.

Laboratory grade orthophosphoric acid (85 %) was used as base solution. For paste experiments, the acid was diluted to different concentrations (see Chapter 3 and 4).
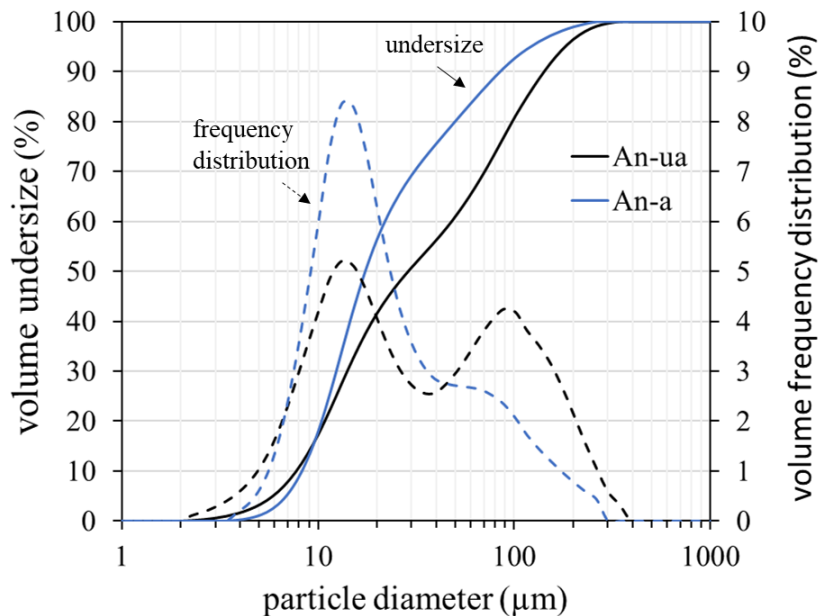
Figure 1 – Particle size distribution of unaltered (An-ua) and altered (An-a) anorthosite

Table 1 – Chemical composition of unaltered (An-ua) and altered (An-a) anorthosite

	XRF (weight %)	
	An-ua	An-a
CaO	14.1	14.0
Al_2O_3	29.9	29.8
SiO_2	47.9	47.5
Fe_2O_3	1.6	1.4
Na_2O	2.8	2.7
K_2O	0.3	0.3
MgO	0.9	0.6
SO_3	0.1	0.1
P_2O_5	0.1	0.0
TiO_2	0.2	0.1
SrO	0.1	0.1
LOI (1000ºC)	1.1	2.4
SUM	99.1	99.1

Table 2 – Qualitative mineralogical composition of unaltered (An-ua) and altered (An-a) anorthosite

Main Phases (<90 %)	Plagioclase: Ca-feldspar (labradorite, bytownite, anorthite)
Minor phases (<10 %)	Plagioclase: Na-feldspar (albite, oligoclase, andesine)
	Others: phlogopite, biotite, chamosite, zoisite, paragonite, montmorillonite, hornblende, pyroxenes, epidote

PROJECT NO.
102019565

REPORT NO.
2021:00335

VERSION
1

6 of 15

3 Experimental

Particle size distribution (PSD) of the materials was measured by laser granulometric analysis with a Horiba Partica LA-960 instrument. Powders were dispersed in water in combination with ultrasound treatment for 1 min to deagglomerate particles.

The chemical composition of the materials was determined by *X-Ray fluorescence analysis (XRF)* using a Bruker AXS S8 Tiger WDXRF. Dried powder was mixed with lithium borate and lithium iodide to produce a homogeneous glass disk for bulk analysis.

The mineralogical composition of the raw materials and hydrated pastes was determined by *X-Ray powder diffraction (XRD)* combined with *Rietveld Analysis*. Samples were prepared by the front-loading technique and measured with Bruker D8 Advance equipped with a LynxEye detector and Cu-Kα X-Ray source.

Thermogravimetric analysis (TGA) was performed as a supplement to XRD. Samples were analysed with a Mettler Toledo TG/SDTA 851 under nitrogen atmosphere in the temperature range 40-900 °C.

Pastes were prepared by mixing anorthosite with diluted phosphoric acid. The concentration of phosphoric acid was varied between 3-15 molar. The ratio between phosphoric acid and anorthosite (PA/An) was varied between 0.3 to 0.5. Pastes were mixed by hand in a plastic cup for about 2 min and stored under sealed conditions for up to 28 days.

In a first test series (screening), the pastes were hydrated at room temperature in a sealed plastic cup to get an indication about the mixing and hardening behaviour. In a second test series the effect of temperature was investigated additionally. Phosphoric acid was heated to the boiling point right before adding it to the anorthosite powder. After mixing, the paste was transferred to small cylindrical glass containers (45x25 mm) for later strength testing. The samples were hydrated under sealed conditions (to avoid drying out) in a heating cabinet at 50, 70, 80 and 90 °C for 28 days. As reference, a fly ash cement (CEM II/B-M 42.5 R, Standard FA, Norcem) paste was prepared with a water to cement ratio of 0.5 and hydrated at room temperature. After 28 days curing, the glass was carefully removed from the hardened samples and the top and bottom surface of the cylinder was polished to get an even surface. The cylinders were tested for *uniaxial strength* in accordance with NS-EN 12390-3.

4 Results and Discussion

4.1 Experiments at room temperature

In a first test series, both anorthosite samples were mixed with different concentrations of diluted orthophosphoric acid (PA) and cured at room temperature. The aim was to visually assess the workability (mixing behaviour) of the different blends and the effect of acid concentration on the strength development. The actual strength was not measured here, only manually assessed by the resistance to penetration with a metal pin/spatula.

The first blends were mixed with a PA/An ratio of 0.4. As this turned out to yield satisfying consistency, the mixing ratio was taken as the basis for the residual blends. However, some mixes were too dry and extra water was needed to obtain a proper consistency. The composition of the blends and observations regarding the consistency are summarized in Table 3.

In general, blends with the altered anorthosite (An-a) had a higher "water" (diluted PA) demand compared to blends with the unaltered anorthosite (An-ua). To enable proper mixing of the powder samples and to guarantee a sufficient degree of reaction extra water had to be added to some mixes. The higher the concentration, a faster reaction and an apparent higher water demand was observed. Blends with An-a had an apparent faster reaction than blends with An-ua (heat development) and therefore also a higher water demand already at relatively low concentrations of PA, as it can be seen in the amounts of extra added water to enable good mixing.

All mixes were setting relatively fast within the first 5-10 min after mixing. The setting time was shorter the higher the concentration of PA. However, none of the mixes obtained a satisfactory strength of practical meaning within the first 14 days, i.e. the blends were easy to penetrate/mark with a metal spatula (Figure 2). The strength was ≤ 1 MPa measured with a PEN penetrometer. After curing for 1-2 month, blends mixed with a 5 and 7 molar PA had an apparent higher strength compared to the rest of the samples (1.5-2 MPa measured with the PEN penetrometer).

The XRD and DTG of the hydrated paste 5a is shown in Figure 3. Comparing the XRD of the unreacted An-ua to the reacted paste, a new peak around 7.5 °2θ was detected. The phase identified for this peak was a Ca-phosphate hydrate ($H_4Ca(PO_4)_2(H_2O)$) probably resulting from the acid leaching of Ca from An and the reaction with phosphate from PA. The formation of hydration phases can also be seen from the TG/DTG curve of the hydrated paste. Weight loss at temperatures above 100 ℃ is due to chemically bound water or hydroxyl ions in the structure of hydration products. The DTG curve shows three distinctive weight losses at around 150, 250 and 350 ℃. The total weight loss is about 9 %. The unreacted anorthosite had a weight loss of only about 1 % (Table 1).

Table 3 – Composition and observation on consistency of test series 1 with blends prepared and cured at room temperature

No.	An-ua (g)	An-a (g)	PA (g)	PA/An [a]	Conc. [a] (mol)	Extra water (g)	Visual observations
1a	20	-	8	0.4	3	-	Normal consistency, easy to mix
1b	-	20	8	0.4	3	-	Higher viscosity compared to the mix with An-ua
2a	20	-	8	0.4	5	-	Normal consistency, easy to mix
2c	-	20	8	0.4	5	1	Dry, extra water needed to obtain similar consistency
3a	20	-	8	0.4	7	-	Less fluid than 1a and 2a, but still easy to mix
3c	-	20	8	0.4	7	1,3	Dry, extra water needed to obtain similar consistency
4a	20	-	8	0.4	10	0,4	Extra water needed to get normal consistency
4b	-	20	8	0.4	10	2,3	Dry, extra water needed to obtain similar consistency
5a	20	-	8	0.4	15	1,8	Dry, extra water needed to obtain similar consistency
5b	-	20	8	0.4	15	3	Dry, extra water needed to obtain similar consistency

[a] PA/An and concentration before adding extra water

Figure 2 – Representative example (5a) of visual appearance of a hardened PA-An paste after 14 days, with spatula marks in the surface

PROJECT NO.
102019565

REPORT NO.
2021:00335

VERSION
1

9 of 15
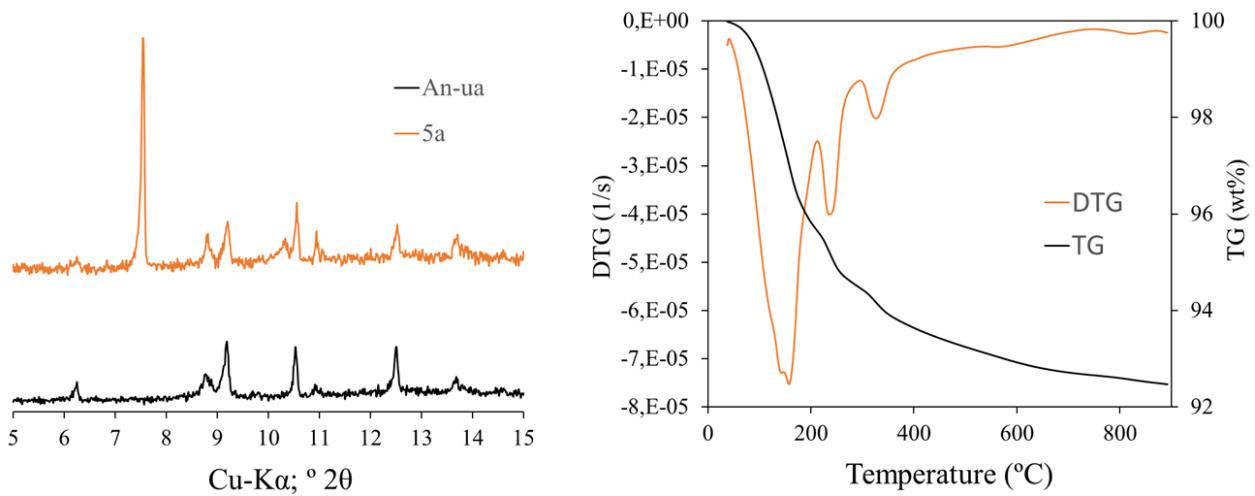
Figure 3 – Left: XRD of unreacted An-ua and paste 5a after 14 days curing; Right: TGA/DTG of paste 5a after 14 days curing.

## 4.2 Experiments with elevated temperature

Although, samples mixed with 15 molar PA showed the fastest strength development, samples mixed with 5 and 7 molar PA had an apparent highest strength after curing for 28-56 days. It was therefore decided to continue in this range of PA concentration. However, the solubility of the anorthosite was probably not high enough at room temperature to result in chemical reactions leading to satisfactory strength development. It is known that the acid solubility (leaching of Al_2O_3) of anorthosite from the Sogn region can be increased with increasing temperature [22].

To accelerate and increase the acid solubility of anorthosite, further tests were therefore performed at different curing temperatures. The second test series was only performed with the unaltered anorthosite sample as it had an apparent higher degree of reaction and better strength development. This might be associated to the reported higher content in anorthite which directly influences the acid solubility [21]. Tests were made with 5 and 7 molar PA and curing temperatures of 50, 70, 80 and 90 °C. Samples were cured in sealed glass containers for 28 days. The ratio PA/An was between 0.35-0.45 and adjusted to achieve an apparent similar consistency of all samples during mixing.

In general, all samples mixed with warm PA had a better flow and were easier to mix compared to samples prepared with cold PA (room temperature). Furthermore, all samples had an apparent higher strength after 1 day than samples prepared at room temperature but cured for at least 28 days. Clearly the higher temperature of the acid helped to activate and increase the dissolution of anorthosite to get increased chemical binding upon curing. Up to 70 °C, all samples were compact in the glass container after curing. At temperatures above 80 °C, small horizontal cracks formed in some samples (Figure 4 - Right). Furthermore, it could be observed that the colour of sample became lighter (towards white) in the first days of curing the higher the curing temperature was (Figure 4). However, after longer curing the colour was similar (light grey – white) in all samples (Figure 5).

Table 4 – Samples of test series 2 to be tested for uniaxial strength

No.	An_ua (g)	PA (g)	PA/An [a]	Conc. [a] (mol)	Curing temperature (ºC)
5M-50C	60	21	0.35	5	50
7M-50C	60	24	0.4	7	50
5M-70C	60	24	0.4	5	70
7M-70C	60	24	0.4	7	70
5M-80C	60	24	0.4	5	80
7M-80C	60	27	0.45	7	80
5M-90C	60	24	0.4	5	90
7M-90C	60	27	0.45	7	90
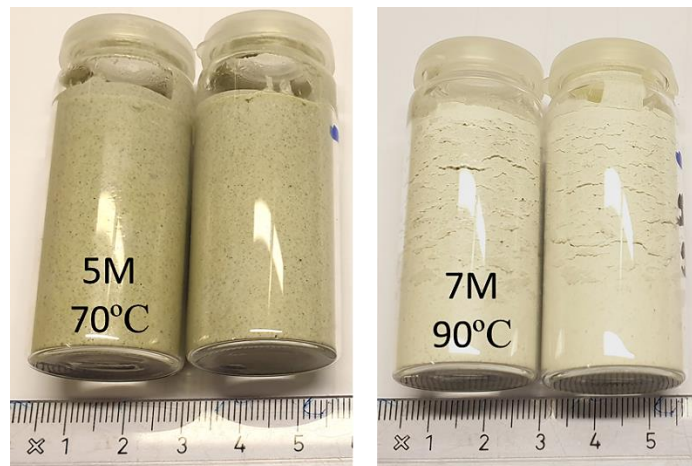
Figure 4 – Representative example of the appearance of different samples; Left: example of "compact" greyish sample cured at 70 ºC, right: Example of a white sample cured at 90 ºC and developing horizontal cracks.


Figure 5 – Colour of samples after compressive strength testing and storage in plastic bags for several months

The strength results after 28 days curing is shown in Figure 6. Some samples reached a strength between 15-20 MPa which can be high enough for certain applications. The strength was about 50 % less than that of the hardened reference cement paste. There is clear trend that higher strength is obtained with increased concentration of PA probably due to increased formation of calcium phosphate hydrates. Strength is relatively stable up to a curing temperature of 70 °C and decreases at higher temperatures. This might be explained by the formation of horizontal cracks in the samples cured above 80 °C.

It is emphasized that these are only preliminary results obtained from a short-term project. The mix and curing design are far from optimized but it was outside the scope of the current project to test further samples. It seems that higher concentration of PA might improve the strength when curing at elevated temperatures. The project group from Canada doing experiments on white mountain anorthosite from Greenland reported strength comparable to ordinary cement. This might be due to the higher purity of the Greenland anorthosite and the higher anorthite content in total. The higher anorthite content of the material from Greenland results in an increased acid solubility, i.e. leaching of Al_2O_3 and other elements [21].

Figure 7 shows TG/DTG curves of pastes prepared with 7 molar PA and cured for 28 days at 50, 70 and 80 °C. In contrast to the paste cured at room temperature only one single weight loss can be observed at around 110 °C with a shoulder between 130-150 °C. On the other hand, the total weight loss was with 13-14 % higher than in the sample cured at room temperature. This might indicate a higher degree of reaction by increased water binding. In contrast to the paste hydrated at room temperature, XRD did not show any new crystalline reaction products. This could indicate the formation of amorphous products, e.g. Ca-Al-Si phosphates formed. An investigation of the microstructure with scanning electron microscopy could give further insights into reaction mechanisms to explain the binding and hardening of anorthosite/phosphoric acid pastes.
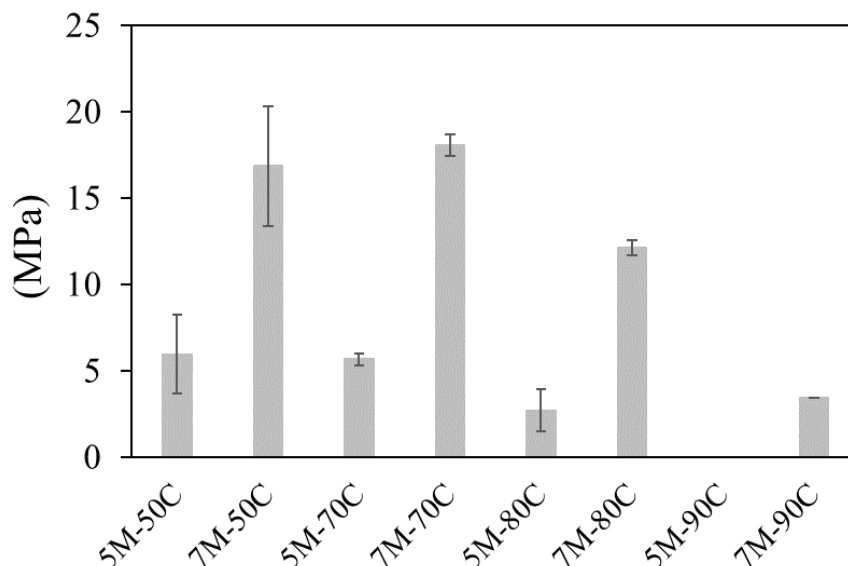
Figure 6 – Strength results of the different mixes after curing for 28 days. Sample 5M-90C cracked before strength testing

PROJECT NO.
102019565

REPORT NO.
2021:00335

VERSION
1

12 of 15
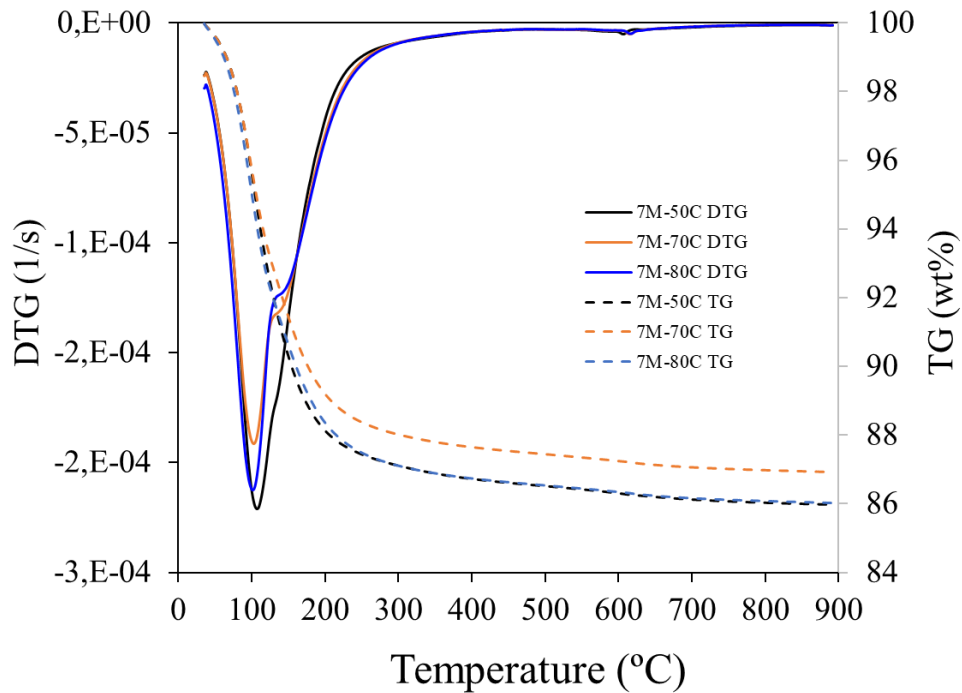
Figure 7 – TG/DTG curves of pastes prepared with 7 molar PA and cured for 28 days at 50, 70 and 80 ºC

5   Conclusions and Perspectives

Norway is rich in natural rock and mineral resources that can enable the production of alternative binders like magnesium phosphate, magnesium hydroxy carbonate cements or alkali activated binders. Additionally, acid activation of rocks might introduce a new concept to produce alternative binders. Although these systems cannot compete with ordinary Portland cement, they might be applicable for certain specialised areas and thus help to reduce the total amount of cement used, consequently reducing the total Norwegian CO_2 emissions. Further research regarding the utilization of Norwegian natural resources in alternative binder concepts is encouraged.

This paper discusses briefly preliminary results of investigations on a binder system based on activation of anorthosite from western Norway with diluted phosphoric acid. Results showed that a strength of close to 20 MPa was reached when mixing anorthosite with 7 molar phosphoric acid and hydrating it at elevated temperature (50-70 ºC) for 28 days.

The study has several limitations and more research is needed to optimize the system and to understand the reactions resulting in the hardened binder. Phosphate is a scarce resource primarily used for agriculture and thus phosphoric acid is not available in large volumes. Other acids, especially waste acid streams should be tested in addition to produce affordable alternative binders.

PROJECT NO.
102019565

REPORT NO.
2021:00335

VERSION
1

13 of 15

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