

CERTIFICATION OF APPROVAL


**Effect of Hydrofluoric Acid on Well Cement during Acidizing**

by

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TRONOH, PERAK

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## CERTIFICATION OF ORIGINALITY

This is to certify that I am responsible for the work submitted in this project, that the original work is my own except as specified in the references and acknowledgements, and that the original work contained herein have not been undertaken or done by unspecified sources or persons.



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CRESCENTIA ESTRALL DIONYSIUS ASAI

## ABSTRACT

This paper covers the study of the effect of hydrofluoric acid (HF) on well cement class G when wells are subjected to acidizing operation. It has been shown that well cement, when exposed to acid attack, will show loss of integrity in providing zonal isolation to the well. Its compressive strength is also affected. Cement samples are cured at a range of pressure and temperature before subjected to acid attack. The effect of acid on the cement samples is quantified in terms of mass loss in cement, decrease in compressive strength and also compositional changes in the cement sample. There are claims that acid attack is purely superficial, where it only attacks the surface of the cement. Further attack is said to be hindered by the formation of a white precipitate identified as fluorite. From the result of the experiment, white precipitate is indeed observed on the surface of the cement cubes exposed to acid attack. To determine whether the attack is confined only to the surface, the sample was sliced open to include the middle portion for x-ray fluorescence (XRF) and scanning electron microscope (SEM) testing. Results show that the middle portion of the cement sample experience little damage to acid attack. For cement cubes cured at higher pressure and temperature, the effect of acid on the well cement is less damaging. We can deduce that pressure and temperature do play a role in determining cement resistance to acid, and that HF attack on cement is superficial.

## **ACKNOWLEDGEMENTS**

This final year project is the culmination of four years of engineering studies in Universiti Teknologi PETRONAS (UTP).

I would like to thank my supervisor, Dr Sonny Irawan for his guidance and support throughout this whole project. His insight helped me stay focused on the objective of this study towards the end.

Many thanks also to the laboratory technicians who helped to prepare materials and equipments, without which running the experiment will not be possible.

Completing this project has helped me develop critical and analytical thinking skills, besides honing my technical writing skills. It is hoped that this study will prove beneficial for those who utilizes it.

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## **ABBREVIATIONS**

SEM	Scanning electron microscope
XRF	X-ray fluorescence
HF	Hydrofluoric acid

## **CHAPTER 1**

### **INTRODUCTION**

Traditional views state that acid reaction with well cement during well acidizing will only occur for a short period of time. The initial belief held is that acid will have little or no effect at all on well cement, with failures attributed mainly to poorly cleaned perforations prior to squeeze cementing and also changes in temperature and pressure while perforating and acidizing the wells. Risk of acidizing causing loss of zonal isolation and breaking down squeezed wells were thought to be minimal. However, field experience proved otherwise. Acid attack towards cement during acidizing has created severe zonal isolation problems in wells.

A significant number of cement squeeze jobs were found to develop zonal isolation problems or broke down when the well is exposed to acidizing. Field data taken from Prudhoe Bay Field, Alaska for example showed that 75% of squeezed wells broke down after being exposed to acid. Compare this with failure rate of only 30% for cement squeezed wells which are not acidized. Solubility of cement in acid is suspected to be the main reason behind this.

This particular project entails the study of well cement resistance to attack of hydrofluoric acid (HF) during acidizing treatment. This involves experimental work to determine the effect of HF in terms of mass of cement, cement composition and compressive strength of cement. Analysis is done before cement is exposed to acid and after exposure. This study is based on the assumption of a clean sandstone formation, which means the formation is completely characterized by sandstone alone. Also assume a neat cement recipe, where cement slurry used is purely class G cement and water.

## 1.1 Background

Part of the process for preparing a well for further drilling, production or well abandonment is well cementing. It involves developing and pumping the cement into place in the wellbore. The ultimate goal and purpose of cementing is to provide zonal isolation, which is a durable seal in the wellbore that allows selective fluids production from the formation. It also aims to prevent leakage from the formation to the well surface as well as among the different zones in the formation, which might affect the purity of the products. Cementing also acts to withstand formation pressure, where cement with a weak compressive strength will result in the collapse of the well, which spells losses in terms of reservoir fluids potential production. Well cement which is already in place is perforated so that reservoir fluids from the formation are able to flow out to the surface. Figure 1 below shows cementing providing zonal isolation for the two different zones in the formation.

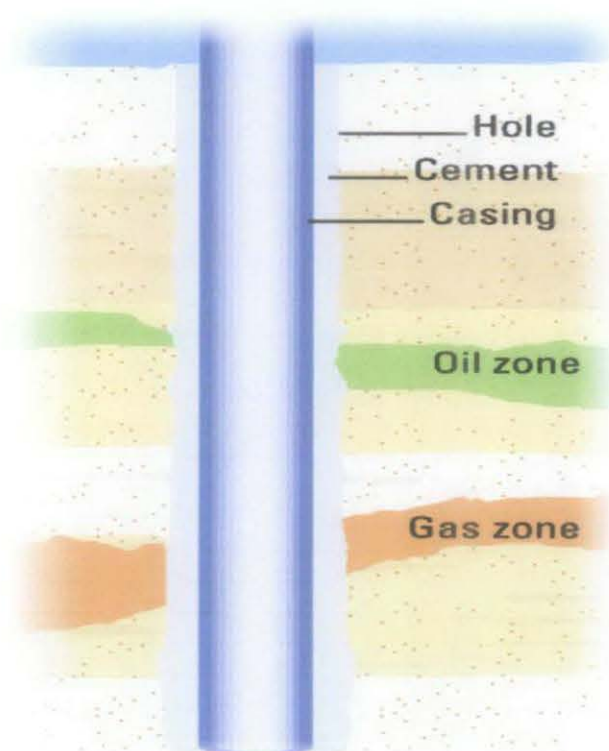


Figure 1: Cementing provides zonal isolation



As the years passed by, production rate might slow down due to clogging of the channels. Stimulation is done to clean the clogged channels and to restore the production rate. Stimulation encourages permeability in the formation so that an underproductive well may experience increase in the flow rate. A type of stimulation is acidizing, where acid is pumped into the well to dissolve sediments which inhibits rock permeability, thus stimulating the flow of hydrocarbons. Figure 2 below shows channels cleaned through acidizing.



Figure 2: Acidizing cleans clogged channels

While acidizing helps to open up channels and increase flow of reservoir fluids, it has an adverse effect on well cement, affecting its ability to provide zonal isolation and also withstanding formation pressure. The degradation on the quality of cement takes place upon first contact with the acid.

This project aims to look at the effects of HF acid attack on well cement, by assuming the HF is used for acidizing a clean sandstone formation and the cement used does not incorporate any additives in it. The curing pressure and temperature of

the cement, which signifies the wellbore condition, is varied to study how these variables affect quality of well cement when exposed to acid attack. Quality of well cement refers to mass loss, compressive strength and compositional changes in the said cement.

## **1.2 Problem Statement**

Acidizing operation cleans out the clogs in the formation behind the well cement to stimulate flow of oil or gas, thus increasing the rate of production. However the downside of this process is that it might affect the well cement affecting the ability of the cement in providing zonal isolation, increasing the chances of different zones interacting with each other.

Another effect of acidizing on well cement is decreasing the mass of the cement as well as the area of the well covered in cement. This will ultimately affect the compressive strength of the cement, risking the possibility of well collapse as the cement can no longer withstand the formation pressure.

This paper will attempt to explain the effect of different curing temperature and pressure of well cement on the cement ability to withstand acid attack. The extent of acid attack will be quantified in terms of mass loss, compressive strength and compositional changes in cement.

## **1.3 Objectives and Scope of Study**

This project aims to:

Evaluate the effect of curing temperature and pressure on the quality of well cement when exposed to acid from acidizing operation, through the analysis of mass loss, compressive strength and composition of cement.

#### **1.4 Project Relevancy**

This project is highly useful in oil and gas field, as acidizing is done in almost every well to stimulate well flow and to increase production rate. By identifying the parameters which might aggravate the effect of acid on the well cement, precautionary actions can be taken to ensure the integrity of the cement is still intact even when exposed to acidizing.

## CHAPTER 2

### THEORY AND LITERATURE REVIEW

#### 2.1 Theory

Cementing is done primarily to provide zonal isolation among the different zones which exist in the formation behind the casing. Zonal isolation means the state or quality where the fluids from a permeable zone are kept separate from the fluids in another permeable zone. Without proper zonal isolation, wells are unlikely to realize their full producing potential. Poor or non-existing cement in the well can contaminate fresh water bearing formation since salt or oil can travel along the casing and ultimately causing damage through the said contamination.

Cementing involves the process of mixing slurry consisting of cement, water and additives. This slurry is then pumped down through steel casing into the annular space between the casing and formation. Hardened cement provides restrictions of fluid flow from different zones in the formation, helps to bond the casing to the formation as well as providing support for the casing. A well can, and has been, lost due to poor cementing job or the non-existence of well cement. One possible scenario involves fresh water travelling up along the casing, dissolving the upper salt layers, leaving behind a huge salt lake instead of a well.

Wells are subjected to high pressure and temperature, which requires specific oil well cement to be used for well cementing. Cement used for well cementing is Portland cement calibrated with additives, which is classified under eight different types according to American Petroleum Institute (API) standards. Each type is used specific to each wellbore condition. The classifications are class A, B, C, D, E, F, G and H. Class G oil well cement is chosen for use in this paper.

Well cementing takes place after casing is placed in a drilled open hole. Cementing head is fixed to the wellhead top to receive slurry pumped from the pumping



equipment. To prevent drilling fluid from mixing with the cement, bottom plug is inserted into the wellbore, where it will sit on top of a float collar situated at the bottom of the wellbore. When introduced into the wellbore, the bottom plug sweeps inside the casing, cleaning the well before cement slurry is pumped in. Float collar acts as a valve which functions as a one-way valve to allow entrance of slurry into the well. Figure 3 below shows the cementing process in a well.

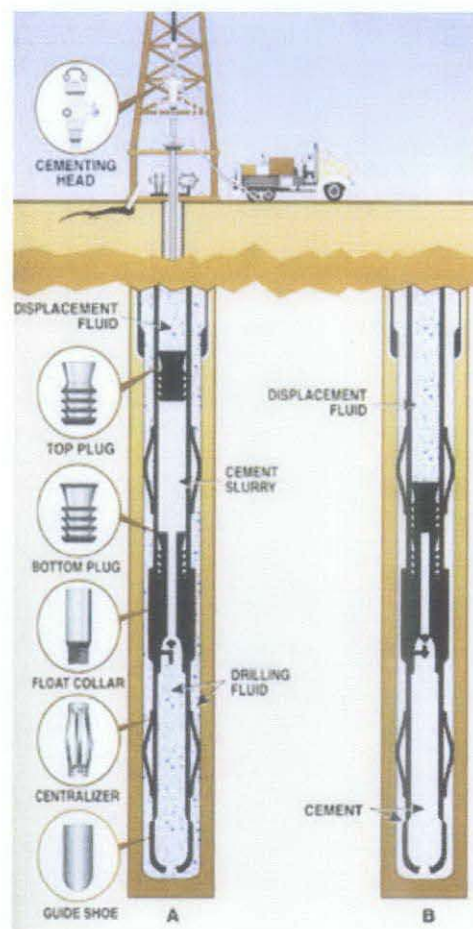


Figure 3: Well cementing (Photo from [www.mpgpetroleum.com](http://www.mpgpetroleum.com))

The diaphragm in the bottom plug is broken when the pressure from the slurry is high enough. This permits the slurry to flow through the bottom plug and outside to the annulus. When the desired amount of cement has been pumped into the well, a top plug is pumped into the casing. This pushes remaining slurry out through the

bottom plug and the top plug will sit on the bottom plug. The cement is then allowed to harden.

Well cementing also provides compressive strength to prevent the casing from collapsing while subjected to formation pressure. Compressive strength refers to the ability of a material to withstand pushing forces directed axially. The material will be crushed once the limit of its compressive strength is met. The compressive strength of cement in this paper is determined using cement compressive strength equipment. The decrease in compressive strength of cement when exposed to acid attack will be calculated using the following equation:

$$\frac{\text{initial compressive strength} - \text{final compressive strength}}{\text{initial compressive strength}} \times 100\%$$

Besides compressive strength, mass of cement will also be affected by the acid attack. Mass loss is calculated as follows:

$$\frac{\text{initial mass} - \text{final mass}}{\text{initial mass}} \times 100\%$$

A loss in mass translates into lower cement density, based on the formula

$$\text{density, } \rho = \frac{\text{mass}}{\text{volume}}$$

A low density means the cement is not tightly packed as before, affecting its strength in withstanding load.

## 2.2 Literature review

There has been very little work on the effect of acid on well cement. When subjected to acidizing treatments, it has been observed that there exists zonal intercommunication problems in the well. The existence of well cement deterioration and the mechanism behind the said process is the reason for this study. Analysis of the cube samples by quantifying the chemical composition changes using the

technique of x-ray fluorescence will yield further understanding on cement solubility in acid solution. The alteration in the chemical composition of the cement and mass loss is also analyzed <sup>[1]</sup>.

Predominantly, the effect of acid on well cement is viewed as superficial and minimized by the formation of protective coating on the well surface which inhibits the continuation of the reaction. However, the effects of acid attack on well interface have been documented using acoustic bond logs before and after the acid attack. It can clearly be seen that following acidization, loss of bond is detected. Even after squeeze cementing to promote re-bonding, subsequent acidizing treatment still result in loss of bond <sup>[2]</sup>.

Several papers have detailed the dissolution of Portland cement by acid solutions; however no methodology has the same specific procedure, making it hard to compare between two experimental results due to discrepancies in the procedure. Some procedure involves keeping HF in glass containers before immersing cement cubes in them. This is a major mistake in the procedure as HF is consumed when attacking the glass, leaving only some amount left for reaction to take place with cement cubes.

Other methods employed include testing cement cubes without properly removing the grease used in the molds beforehand and different concentration and volume of acid used in different sets of experiments. This further complicates comparison of experimental results. This clearly calls for a well defined methodology in ensuring valid and comparative data is obtained. Equipments used for testing are highly detailed to limit technical errors due to difference in configurations and quality.

Safety is a huge concern when dealing with acid, thus it is highly necessary to have the MSDS ready and read before handling the acid. Handle the acid in a fume hood using acid resistant rubber gloves and apron plus safety glasses with side shields. Make sure the workstation is equipped with emergency showers and eyewash station. A lab partner is highly recommended to be present when conducting the experiment as an added safety precaution <sup>[3]</sup>.

## CHAPTER 3

### METHODOLOGY

#### 3.1 Research Methodology

Figure 4 below shows an overview of the experimental procedure.

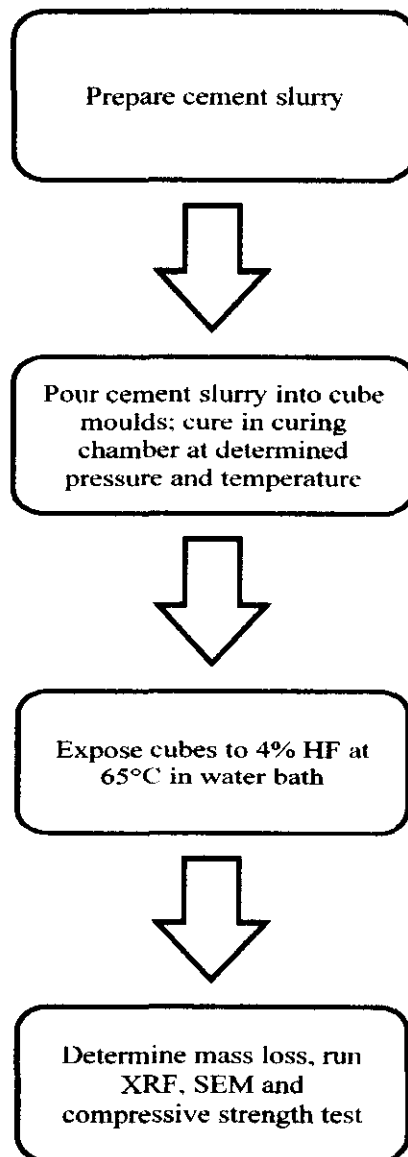


Figure 4: Experimental procedure flow chart



*Detailed experimental procedure:*

**Cement cubes preparation:**

1. 787.09 g of oil well cement class G is poured into 349.08 g fresh water in blending cup in 15 seconds at 4000 rpm. The rotation is then switched to 12000 rpm and left for 35 seconds (Based on API Specification 10A).
2. Cement slurry is poured into cube moulds and cured in curing chamber for eight hours at 3000 psi and 90°F.
3. Only cubes with perfect sides are chosen to be used in the subsequent tests to avoid errors in test results due to imperfect cube surface conditions.
4. Dry the cement cubes and weigh the mass. After that test for compressive strength, XRF and SEM. This will be the initial mass, compressive strength and composition of cement before acid exposure.
5. Repeat steps 1-4 for these curing conditions: 3000 psi and 150°F, 3000 psi and 200°F, 4000 psi and 175°F, 5000 psi and 175°F.

**Acid exposure experimental procedure:**

1. 4% HF is prepared in 5000 ml plastic beaker. Heat acid in water bath until it reaches 65°C.


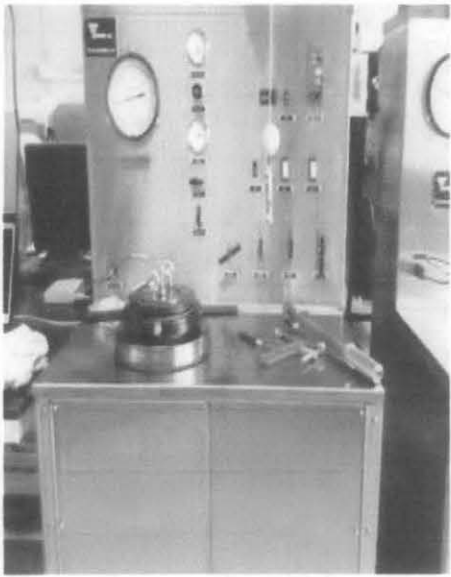
**Warning:** Measure temperature from water inside another plastic beaker placed next to the one containing acid. HF is reactive to glass, including thermometer.

2. Place cement cube into acid and leave for 40 minutes.
3. The cubes are then taken out and left to dry for one hour. Measure the mass and compressive strength of the cement. Test also using XRF and SEM, this time including the middle portion of the sample. This will be the mass, compressive strength and composition of cement after acid exposure.

### 3.3 Tools/Equipment

Table 1 below shows the various tools/equipments used in this project.

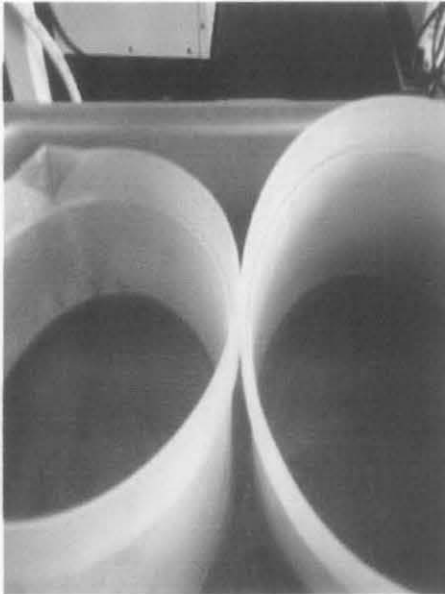
Table 1: Tools/equipments used

Tools/Equipment	Description
	<p>Constant speed mixer</p> <ul style="list-style-type: none"><li>• Mixing cement slurry prior to pouring it into cube moulds</li></ul>
	<p>High pressure, high temperature (HPHT) consistometer</p> <ul style="list-style-type: none"><li>• Curing cement cubes at determined pressure and temperature for eight hours</li></ul>



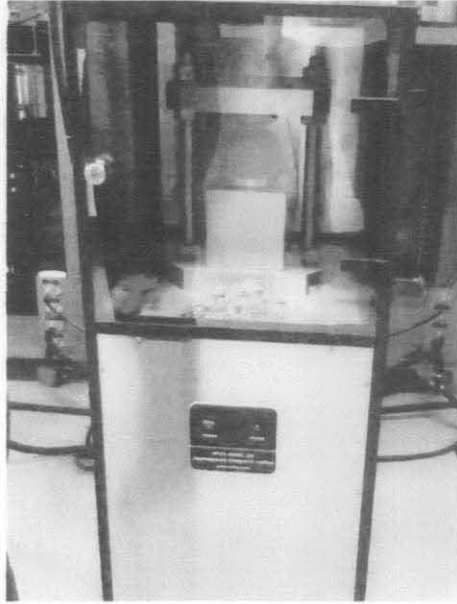
#### Water bath

- Heat acid to 65°C before putting in cement cube for 40 minutes.



#### Polypropylene beaker

- One contains 4% HF, while the other one contains water for temperature measurement



#### Compressive strength tester

- Cement is tested for its maximum compressive strength. Cement is crushed when it reaches its maximum strength.

## CHAPTER 4

### RESULT AND DISCUSSION

Figure 5 below shows the images of cement cube before and after acid attack.


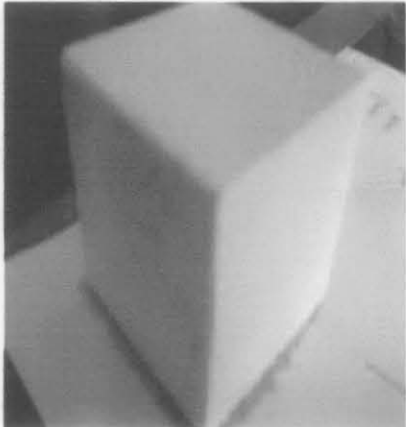
<i>Before acid attack</i>	<i>After acid attack</i>
	
<b>Remarks:</b> White precipitate can be seen on the surface of the attacked cube.	

Figure 5: Cement sample before and after acid exposure

#### 4.1 Mass loss

Table 2 below shows the mass loss for cement cubes cured at constant pressure of 3000 psi:

Table 2: Mass loss (%) at constant pressure of 3000 psi

<i>Temperature (°F)</i>	<i>Mass before acid attack (g)</i>	<i>Mass after acid attack (g)</i>	<i>Mass loss (%)</i>
90	115.97	112.28	3.18
150	111.94	108.50	3.07
200	106.29	104.50	1.68

Figure 6 below shows the graph for mass loss (%) for cement cured at constant pressure of 3000 psi.

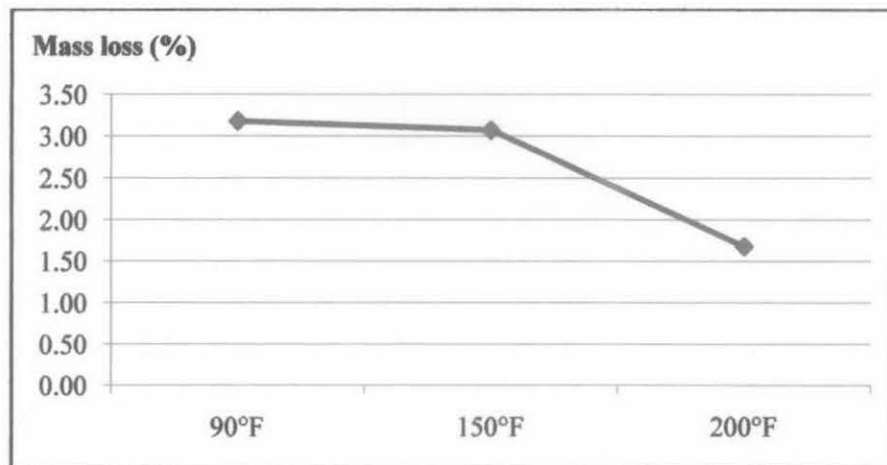


Figure 6: Mass loss (%) graph at constant pressure of 3000 psi.

From the graph, it is observed that as curing temperature increases, mass loss becomes lesser.

Table 3 below shows the mass loss for cement cubes cured at constant temperature of 175°F:

Table 3: Mass loss (%) at constant temperature of 175°F

<i>Pressure (psi)</i>	<i>Mass before acid attack (g)</i>	<i>Mass after acid attack (g)</i>	<i>Mass loss (%)</i>
3000	111.25	109.10	1.93
4000	109.12	107.90	1.12
5000	105.93	104.80	1.07

Figure 7 below shows the graph for mass loss (%) for cement cured at constant temperature of 175°F:

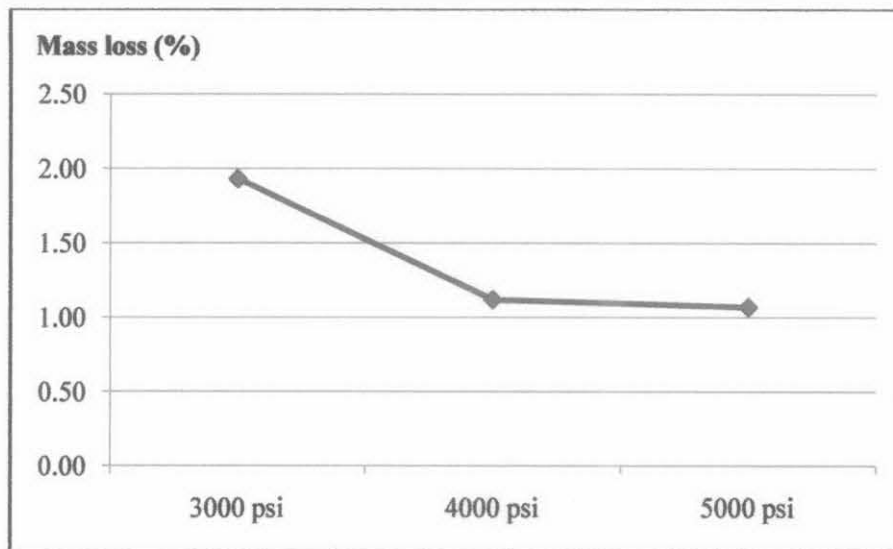


Figure 7: Mass loss graph at constant temperature

From the graph, it is observed that as curing pressure increases, mass loss decreases.

## 4.2 Compressive strength

Table 4 below shows the decrease in compressive strength for cement cubes cured at constant pressure of 3000 psi:

Table 4: Compressive strength loss (%) at constant pressure

<i>Temperature (°F)</i>	<i>CS before acid attack (psi)</i>	<i>CS after acid attack (psi)</i>	<i>Decrease in CS (%)</i>
90	3334	3096	7.14
150	3596	3439	4.37
200	3879	3789	2.32

Figure 8 below shows the graph for compressive strength loss (%) for cement cured at constant pressure of 3000 psi.

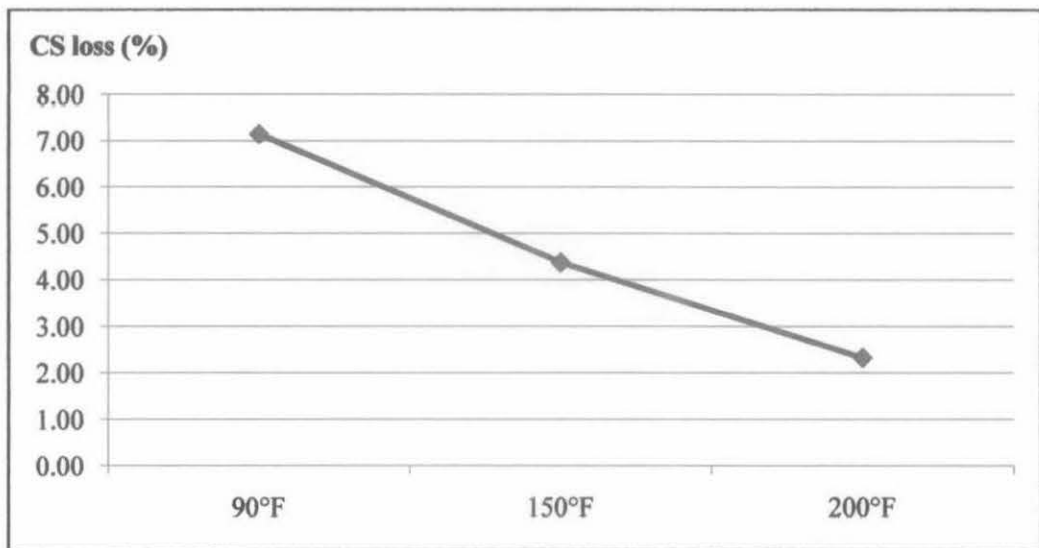


Figure 8: Compressive strength loss (%) graph at constant pressure

From the graph, it is observed that as curing temperature increases, decrease in compressive strength becomes lesser.



Table 5 below shows the decrease in compressive strength for cement cubes cured at constant temperature of 175°F:

Table 5: Compressive strength loss (%) at constant temperature

<i>Pressure (psi)</i>	<i>CS before acid attack (psi)</i>	<i>CS after acid attack (psi)</i>	<i>Decrease in CS (%)</i>
3000	6350	6104	3.87
4000	6739	6597	2.11
5000	6975	6867	1.55

Figure 9 below shows the graph for compressive strength loss (%) for cement cured at constant temperature of 175°F.

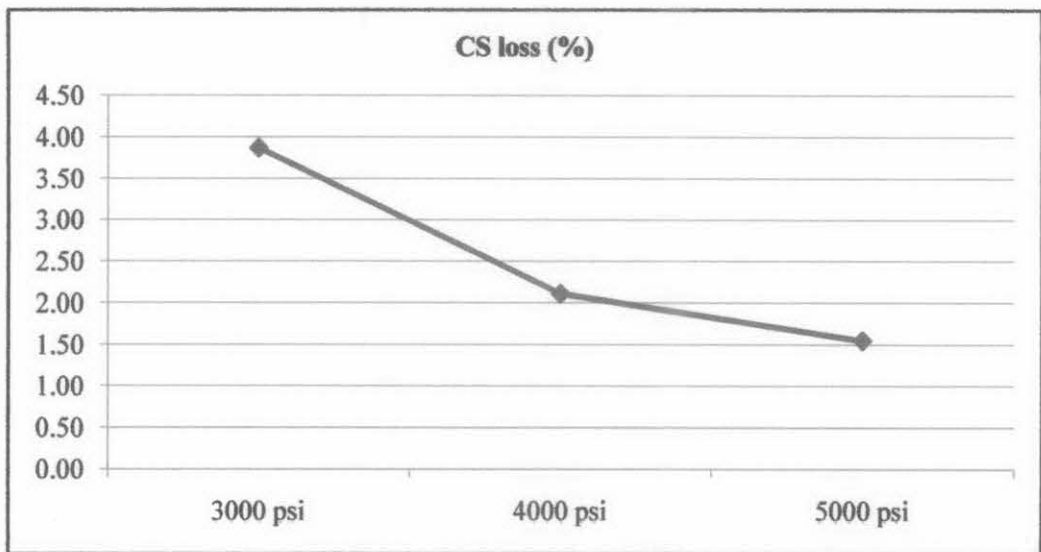


Figure 9: Compressive strength loss (%) graph at constant temperature

From the graph, it is observed that as curing pressure increases, decrease in compressive strength becomes lesser.

### 4.3 X-ray fluorescence (XRF)

We will look into the quantitative changes in cement composition before and after acid exposure.

Table 6 below shows the XRF result for cement sample cured at 3000 psi, 90°F:

Table 6: XRF for cement cured at 3000 psi, 90°F

Component	Before acid exposure (%)	After acid exposure (%)	
		Surface	Middle
MgO	0.92	0.97	0.945
Al <sub>2</sub> O <sub>3</sub>	2.93	3.54	2.64
SiO <sub>2</sub>	24.6	24.0	22.0
SO <sub>3</sub>	1.63	1.38	1.49
K <sub>2</sub> O	0.065	0.996	0.324
CaO	63.89	62.65	66.17
Fe <sub>2</sub> O <sub>3</sub>	4.057	3.78	4.650

Table 7 below shows the XRF result for cement cured at 3000 psi, 200°F:

Table 7: XRF for cement sample cured at 3000 psi, 200F

Component	Before acid exposure (%)	After acid exposure (%)	
		Surface	Middle
MgO	0.92	0.93	0.85
Al <sub>2</sub> O <sub>3</sub>	2.60	2.33	2.45
SiO <sub>2</sub>	23.1	18.3	19.3
CaO	64.51	69.78	69.51
Fe <sub>2</sub> O <sub>3</sub>	4.708	4.965	4.796

Table 8 below shows the XRF result for cement cured at 3000 psi, 175°F:

Table 8: XRF for cement sample cured at 3000 psi, 175°F

Component	Before acid exposure (%)	After acid exposure (%)	
		Surface	Middle
MgO	0.79	1.0	0.93
Al <sub>2</sub> O <sub>3</sub>	2.38	2.36	2.44
SiO <sub>2</sub>	22.1	19.4	20.0
K <sub>2</sub> O	0.045	0.127	0.221
CaO	67.01	68.86	68.50
Fe <sub>2</sub> O <sub>3</sub>	4.618	4.833	4.724

Table 9 below shows the XRF result for cement cured at 5000 psi, 175°F

Table 9: XRF for cement sample cured at 5000 psi, 175°F

Component	Before acid exposure	After acid exposure (%)	
		Surface	Middle
MgO	0.875	1.02	0.95
Al <sub>2</sub> O <sub>3</sub>	2.38	2.29	2.18
SiO <sub>2</sub>	21.68	20.1	19.8
K <sub>2</sub> O	0.083	0.192	0.467
CaO	67.45	68.52	68.40
Fe <sub>2</sub> O <sub>3</sub>	4.827	4.789	5.122

#### 4.4 Scanning Electron Microscope (SEM)

Figure 10 below shows the initial SEM image for a cement sample yet to be exposed to cement is as below:

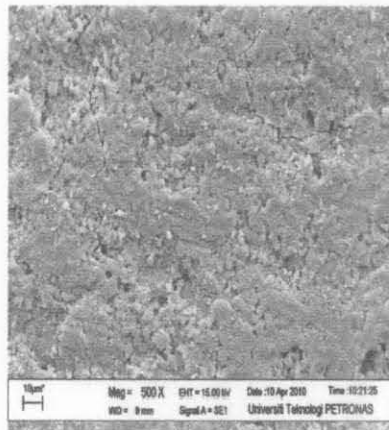


Figure 10: Initial SEM

Table 10 shows the SEM images for cement sample cured at constant pressure of 3000 psi and exposed to acid attack.

Table 10: SEM for cement sample cured at constant pressure

<i>Temperature (°F)</i>	<i>Surface sample</i>	<i>Middle sample</i>
90		
Remarks	Crack is observed at the surface.	Crack is no longer observed in the middle of the sample.

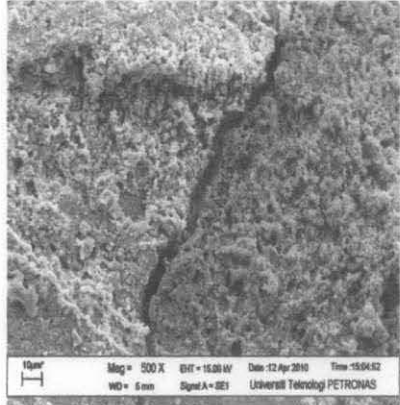
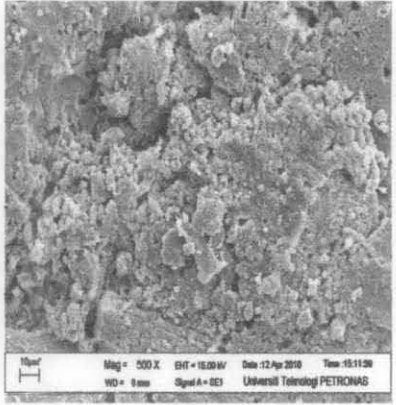
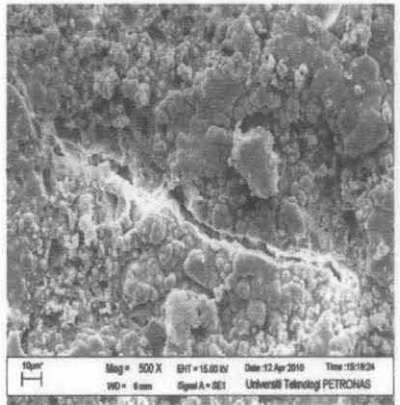
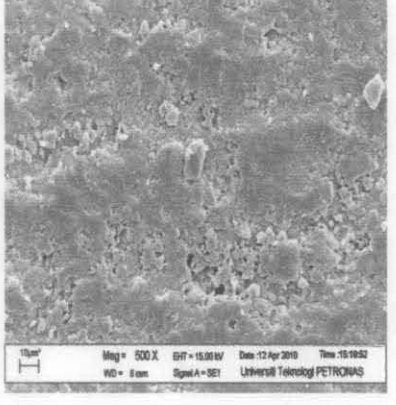
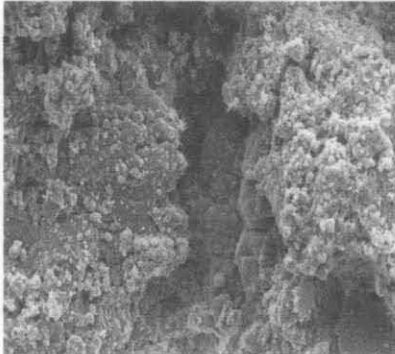
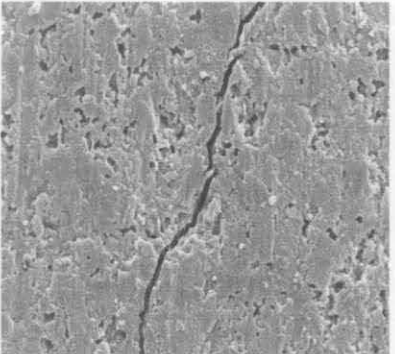
200		
Remarks	Crack is observed at the surface.	Crack is no longer observed in the middle of the sample.

Table 11 below shows the SEM images of cement sample cured at constant temperature of 175°F and exposed to acid attack.

Table 11: SEM for cement sample cured at constant temperature

<i>Pressure (psi)</i>	<i>Surface sample</i>	<i>Middle sample</i>
3000		
Remarks	Crack is observed at the surface.	Crack is no longer observed in the middle of the sample.

5000	 <p data-bbox="500 719 898 763"> <small>Mag = 500 X EHT = 15.00 kV Date = 12 Apr 2010 Time = 15:28:35  WD = 8 mm Signal A = SE1 Universiti Teknologi PETRONAS</small> </p>	 <p data-bbox="932 719 1330 763"> <small>Mag = 500 X EHT = 15.00 kV Date = 12 Apr 2010 Time = 15:31:19  WD = 8 mm Signal A = SE1 Universiti Teknologi PETRONAS</small> </p>
Remarks	Deep crack observed at surface.	Smaller crack observed in the middle.

#### 4.5 Discussion

For mass loss and compressive strength, it can be seen that as curing pressure and temperature goes higher, mass loss and compressive strength loss gets lesser after being exposed to acid attack. We can deduce that higher curing pressure and temperature lends an effect in increasing the strength of the cement.

For both conditions, at constant pressure of 3000 psi and constant pressure of 175°F, we can see that cracks are more dominant on the surface of sample compared to the middle sample. This suggests that acid attack is a surface occurrence <sup>[1]</sup>.

It is observed that acid-exposed cement has a white layer surrounding it. Interactions between cement and acid result in this white precipitation which is identified as fluorite. This white precipitate might be the reason why acid attack is mainly a surface phenomenon. The white layer impedes further attack on the inside layer, thus defending the cement from further damage.

XRF result shows the presence of elements such as magnesium, aluminum, potassium, sulfur, calcium and silicon. These materials became soluble when exposed to acid, thus resulting in mass loss <sup>[1]</sup>. This can also explain the decrease in compressive strength after acid exposure, as mass loss reduces the density of the cement, which interferes with its ability to withstand load applied to it.

It is also observed that the percentage of SiO<sub>2</sub> is considerably lower at the surface of the attacked cubes, signifying reaction with acid. The middle sample shows almost equivalent amount of SiO<sub>2</sub> with the unattacked cubes, signifying acid attack is superficial <sup>[1]</sup>. The actual result obtained may not necessarily reflect this due to contamination of the sample prior to testing. This can be caused by improper storage of the samples.

A large concentration of iron (III) is seen based on the XRF result due to interactions between acid and cement. It helps to stabilize acid-in-oil emulsions which reduce well productivity <sup>[4]</sup>.

Samples cured at high pressure (3000 psi and above) show an evident reduction in acid solubility. This is because curing at high pressure minimizes cement permeability. Cement acid solubility test (CAST) results are qualitative. 100%

solubility in acid cannot be achieved due to small volume of acid used compared to the huge cement sample size. Cement samples which undergo 20-30% solubility can approach 100% solubility given greater acid volume and exposure time. It is more accurate to point out that acid attack is retarded and not prevented when acid solubility exceeds 8-10%.

All these test procedures and results only provide a solubility range for a given test conditions without coming up with a constant which encompasses an absolute solubility value. When using the term “acid-resistant” for cement samples developed to withstand the effect of acid during acidizing treatment, it does not necessarily mean that they are acid proof.

If the volume of acid used is increase to >4000ml and the time of exposure is lengthen, a more quantitative results can be obtained. In the longer term, however, this can translate into safety issues and equipment problems. An alternative to this is by using smaller cement sample to compensate for the small volume of acid used. This smaller sample mass means calculation values for weighing, drying and others will be magnified, contributing to errors<sup>[3]</sup>.



## **CHAPTER 5**

### **CONCLUSION & RECOMMENDATION**

#### **5.1 Conclusion**

1. Curing pressure and temperature play roles in determining whether cement has the capacity to withstand outside influences without little or no damage at all. Higher curing temperature and pressure result in less mass loss and less compressive strength loss.
2. It is also observed that acid attack is mainly a surface phenomenon, attacking on cement surface with little or no damage at all to the inner layers. After acid exposure it is noted that the sample is covered with white precipitate, which seems to impede further damage by the acid on the inner layers of the sample.

#### **5.2 Recommendation**

1. A longer curing period will ensure proper strength development.
2. Ensure cement cubes are thoroughly cleaned from lubricants used in the moulds. Lubricant layer will act as a diffusion barrier, hindering contact between acid and cement.
3. Use only plastic containers when dealing with HF. HF will react with glass, reducing the strength of acid used for testing.
4. Polish the surface of the cement sample for a more accurate imaging.
5. Test for compressive strength as soon as sample is prepared as strength develops over time.
6. Keep exposed cement sample in air tight containers prior to XRF testing, as factors such as humidity will affect the test result.

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## APPENDIX

### *Scanning electron microscopy (SEM)*

Takes images of the sample surface by scanning it with high-energy electron beams.  
Identifies the sample surface topography, composition and other properties.

### *X-ray fluorescence (XRF)*

Analyze the percentage of elements present in the cubes before and after acid attack.

### *Compressive strength press*

Identify the compressive strength of the cement by placing load on the cube surface until the cube fractures.

### *Acid preparation:*

The supplied acid is 48%-50% in concentration; however the required concentration is 4% with a volume of 3 L. For dilution of acid:

$$M_1 V_1 = M_2 V_2$$

where  $M_1$  = initial molar concentration

$V_1$  = initial volume

$M_2$  = final molar concentration

$V_2$  = final volume

Sample calculation for 4% acid:

$$(48) V_1 = (4) (3)$$

$$V_1 = 0.25 \text{ L}$$

This means that 0.25 L of 48%-50% of acid is required for dilution to 3 L of 4% acid.

*Sample calculation:*

Average mass of cement before acid exposure (taken as initial mass)

$$= \frac{\text{mass of cube 1} + \text{mass of cube 2}}{2}$$

$$= \frac{116.33 + 115.61}{2}$$

$$= 115.97$$

Mass of cement after acid exposure (taken as final mass)

$$= \frac{\text{mass of cube 1} + \text{mass of cube 2}}{2}$$

$$= \frac{112.81 + 111.74}{2}$$

$$= 112.28$$

Mass loss (%)

$$= \frac{\text{initial mass} - \text{final mass}}{\text{initial mass}} \times 100\%$$

$$= \frac{115.97 - 112.28}{115.97} \times 100\%$$

$$= 3.18\%$$

*Full listing of XRF result:*

XRF for cement cured at 3000 psi, 90°F

Component	Before acid exposure (%)	After acid exposure (%)	
		Surface	Middle
MgO	0.92	0.97	0.945
Al <sub>2</sub> O <sub>3</sub>	2.93	3.54	2.64
SiO <sub>2</sub>	24.6	24.0	22.0
P <sub>2</sub> O <sub>5</sub>	1.56	2.52	1.34
SO <sub>3</sub>	1.63	1.38	1.49
K <sub>2</sub> O	0.065	0.996	0.324
CaO	63.89	62.65	66.17
TiO <sub>2</sub>	0.16	-	0.15
MnO	0.061	-	0.066
Fe <sub>2</sub> O <sub>3</sub>	4.057	3.78	4.650
CuO	0.0647	0.0731	0.0733
ZnO	0.0899	0.0724	0.105

XRF for cement sample cured at 3000 psi, 200F

Component	Before acid exposure (%)	After acid exposure (%)	
		Surface	Middle
MgO	0.92	0.93	0.85
Al <sub>2</sub> O <sub>3</sub>	2.60	2.33	2.45
SiO <sub>2</sub>	23.1	18.3	19.3
P <sub>2</sub> O <sub>5</sub>	2.45	1.94	1.22
SO <sub>3</sub>	1.43	1.29	1.28
CaO	64.51	69.78	69.51
Fe <sub>2</sub> O <sub>3</sub>	4.708	4.965	4.796
CuO	0.130	0.094	0.0718
ZnO	0.137	0.111	0.106

XRF for cement sample cured at 3000 psi, 175°F

Component	Before acid exposure (%)	After acid exposure (%)	
		Surface	Middle
MgO	0.79	1.0	0.93
Al <sub>2</sub> O <sub>3</sub>	2.38	2.36	2.44
SiO <sub>2</sub>	22.1	19.4	20.0
P <sub>2</sub> O <sub>5</sub>	1.17	1.57	1.38
SO <sub>3</sub>	1.43	1.37	1.39
K <sub>2</sub> O	0.045	0.127	0.221
CaO	67.01	68.86	68.50
TiO <sub>2</sub>	0.14	0.15	0.15
MnO	0.066	0.06	0.059
Fe <sub>2</sub> O <sub>3</sub>	4.618	4.833	4.724

CuO	0.0789	0.0953	0.0746
ZnO	0.126	0.130	0.125
SrO	0.022	-	-

XRF for cement sample cured at 5000 psi, 175°F

Component	Before acid exposure	After acid exposure (%)	
		Surface	Middle
MgO	0.875	1.02	0.95
Al <sub>2</sub> O <sub>3</sub>	2.38	2.29	2.18
SiO <sub>2</sub>	21.68	20.1	19.8
P <sub>2</sub> O <sub>5</sub>	0.812	1.20	1.36
SO <sub>3</sub>	1.43	1.45	1.28
K <sub>2</sub> O	0.083	0.192	0.467
CaO	67.45	68.52	68.40
TiO <sub>2</sub>	0.161	0.15	0.15
MnO	0.0689	0.0672	0.056
Fe <sub>2</sub> O <sub>3</sub>	4.827	4.789	5.122
CuO	0.0738	0.0753	0.0829
ZnO	0.125	0.117	0.120
SrO	0.0264	0.0310	-

## GANTT CHART

No.	Detail/ Week	1	2	3	4	5	6	7	8	9	10	11	12	13	14	15	16	17	18	19	20	
1	Briefing & update on students progress								Mid-semester break													
2	Project work commences																					
3	Submission of Progress Report 1																					
4	Submission of Progress Report 2																					
5	Poster Exhibition/Pre-EDX																					
6	EDX																					
7	Submission of Final Report																					
8	Final Oral Presentation																					
9	Submission of hardbound copies																					