Chapter 3

Preparation of samples and evaluation methods

3.1. cement slurry specification

Cement material and mixing of cement slurry have standard preparation designed by global institutes such as an API (American petroleum institute) and ASTM (American society of testing material) those two initiates are well known in oil and gas industry.

3.1.1. Cement classes standards:

Avery class of cement have special standard according to (API spec10A, 2009) this standards show manufacturing standards and properties standards; hear are tables blow she these standards.

Table (3.1): API classes of Portland cement (API spec10A, 2009)

API Class	Compounds, % C_3S	C_2S	C_3A	C_4AF	Wagner Fineness, cm^2/g
A	53	24	8+	8	1,500 to 1,900
B	47	32	5–	12	1,500 to 1,900
C	58	16	8	8	2,000 to 2,800
G & H	50	30	5	12	1,400 to 1,700

Property	How Achieved
High early strength	By increasing the C_3S
Better retardation	By controlling C_3S and C_3A
Low heat of hydration	By limiting the C_3S and C_3A content
Resistance to sulfate attack	By limiting the C_3A content

Table (3.2): typical physical properties of API cement (API spec10A, 2009)

Well cement class:				A	B	C	G	H
Mix water, wt% of well cement:				46	46	56	44	38
Fineness tests (alternative methods):								
Turbidimeter (specified surface, minimum, m_2/kg):				150	160	220	—	—
Air permeability (specified surface, minimum, m_2/kg):				280	280	400	—	—
Free-fluid content, maximum, mL:				—	—	—	3.5	3.5
Compressive-strength test, 8-hour curing time	Schedule number, Table 7	Curing temp., °F (°C)	Curing pressure, psi (kPa)	Minimum Compressive Strength, psi (MPa)				
	—	100 (38)	Atmos.	250 (1.7)	200 (1.4)	300 (2.1)	300 (2.1)	300 (2.1)
	—	140 (60)	Atmos.	—	—	—	1,500 (10.3)	1,500 (10.3)
Compressive-strength test, 24-hour curing time	Schedule number, Table 7	Final curing temp., °F (°C)	Final curing pressure, psi (kPa)	Minimum Compressive Strength, psi (MPa)				
	—	100 (38)	Atmos.	1,800 (12.4)	1,500 (10.3)	2,000 (18.8)	—	—
Pressure/ temperature thickening-time test	Specifi-cation test schedule number, Table 10		Maximum consistency, 15 to 30 min stirring period, B_c	Minimum Thickening Time, min				
	4		30	90	90	90	—	—
	5		30	—	—	—	90	90
	5		30	—	—	—	120 max.	120 max.

B_c = Bearden units of consistency, obtained on a pressurized consistometer, as defined in Sec. 9 of API *Spec. 10A* and calibrated as per the same section.[8]

3.1.2. Mixing and samples standers:

According to(API spec10A, 2009) cement slurry has specific mixing standers , these specification shoe how to mix the slurry , special mixture type and its velocity also the percentage of water used to be mixed. Also it shoes samples preparing, here are the standers:

1. Using of mixing blender device, of capacity 1 liter (1 quart), having bottom drive and a blade-type blender as shown in figure (3.1).

Figure (3.1) common mixing devises (API spec10A, 2009)

1. The room temperature should be $(25^{\circ}-30^{\circ})$ C and (4000±200rpm) devise speed must be used for (15 ±1) Sec with constant rate of water to mix the slurry, after that continue the mixing to (35 ±1) Sec at (12000 ±500 rpm).

2. After mixing the slurry must putted at blocks made from glass or smooth material to make the samples of testing.

3.2. Samples preparation

Cement type which was used to prepare the samples is class g cement because of its usage of Sudan's fields; sample preparation operation takes several steps beginning with dry cement preparation, and mesquite preparation ending with making samples.

3.2.1. Dray cements preparation:

Dry cement was sieved by mish (90µm) to obtain the fine grains also to ensure the cement is empty from the flocculate

Figure (3.2): show the cement during sieving

3.2.2. Mesquite wood powder Preparation:

Mesquite branches was cut in to small parts (5cm), then they were proffered to the sun rays for 3 days to replace any inside wet, then the small branch was powdered by using special powder maker.

Figure (3.3): mesquite branches before milling

Mesquite powder was sieved by the same mish used to dry cement to obtain the same size of cement.

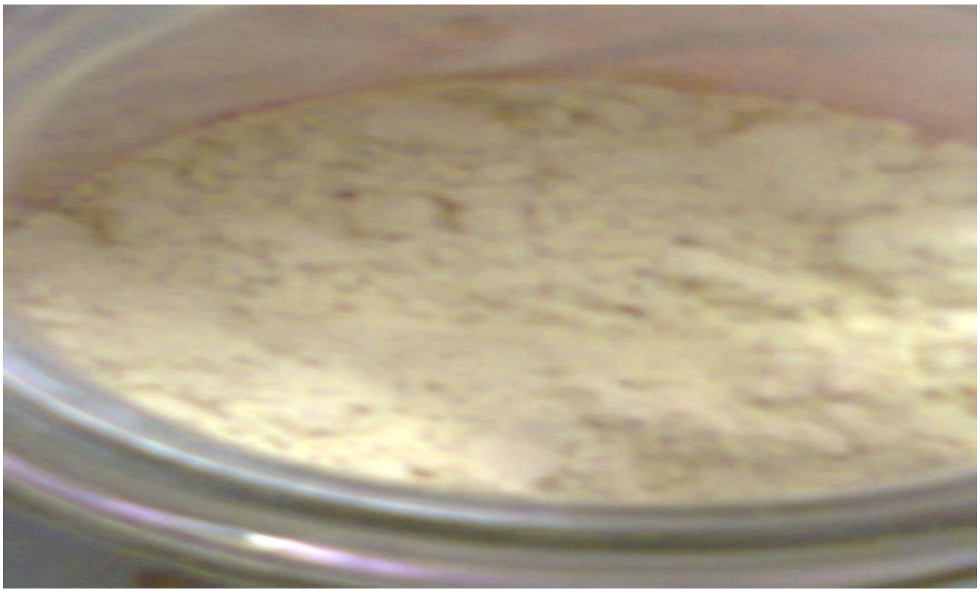

Figure (3.4): mesquite wood powder

3.2.3. Sample preparation:

To prepare the samples 0.308 litter of water was added to 700 gm cement class-G according to API standard, mesquite powder was added with deference percentage to slurry, and then the slurry mixed according to standards.

Five different samples was prepared, the difference between these samples is the quantities of mesquite powder which added to the cement slurry, then they were putted in molds of smooth play wood as shown in figure (3.5) and (3.6).

Figure (3.5): typical smooth play wood

Figure (3.6): the cement sample contain additive

Table (3.3): the percentage of mesquite wood powder added to samples.

Sample No.	**Mesquite powder weight, (gm)**	**Percentage from dry cement, (%)**
1	0	0
2	0.77	0.11
3	1.54	0.22
4	2.31	0.33
5	3.08	0.44

3.3. Evaluation methods

To evaluate the effect of mesquite in preventing micro crack of cement samples several test methods must be used; those test methods are divided into destructive and non-destructive tests.

3.3.1. Destructive tests (DT):

Is the type of test which material has been crushed and being useless. It contains compressive strength.

I. The compressive strength:

In the study of strength of materials, the compressive strength is the capacity of a material or structure to withstand loads tending to reduce size. It can be measured by plotting applied force against deformation in a testing machine. Some material fracture at their compressive strength limit; others deform irreversibly, so a given amount of deformation may be considered as the limit for compressive load. Compressive strength is a key value for design of structures in the Figure (3.7) show compressive strength machine.

In cement the compressive strength of the set cement is the compression force required to crush the cement divided by the cross-sectional area of the sample the stander of compressive strength of cement 440 psi at 60ºF and 24 hours.

Objective of Compressive strength

1. measured stress of concrete
2. specified hardness of concrete
3. measure consistence of concrete

Figure (3.7): show compressive strength machine (Thomas, 2014)

3.3.2. Nondestructive tests (NDT):

I. Inspection radiographic (x ray):

X-rays are part of the electromagnetic spectrum, with wavelengths shorter than light. Most X-rays have a wavelength in the range of 0.01 to 10 nanometers, corresponding to frequencies in the range 30 pet hertz to 30 megahertz (3×10^{16} Hz to 3×10^{19} Hz) and energies in the range 100 eV to 100 keV. However, much higher-energy X-rays can be generated for medical and industrial uses, for example radiotherapy, which utilizes linear accelerators to generate X-rays in the ranges of 6–20 MeV.

X-ray wavelengths are shorter than those of UV rays and typically longer than those of gamma rays. In many languages, X-radiation is referred to with terms meaning Rontgen radiation, after Wilhelm Rontgen, who is usually credited as its discoverer, and who had named it *X*-radiation to signify an unknown type of radiation.

Purpose of X-Ray:

1. Metals inspection.
2. 2-concrate inspection.

II. Easy scanning:

By this technique we can get the picture of atomic resolution, and then we can determine the distances between particles, that can be achieved by device called 'easy Scan' show in figure (3.9).

A small sharp platinum tip show in figure (3.10) is scanned across sample surface, so the tunneling current can flow and with help of this current the tip-surface distance can be controlled very precisely. Therefore an enormous resolution is achieved so that the atomic arrangement of sample surface can be probed.

With easy Scan STM (Scanning Tunneling Microscope) the platinum tip clamped between tow tinny spring and platform which can be moved in all three dimensions. All three axes are driven very precisely nanometer rang. The sample which to be examined is brought close to tip to a distance several nanometers.

Figure (3.8) show the easy Scan device

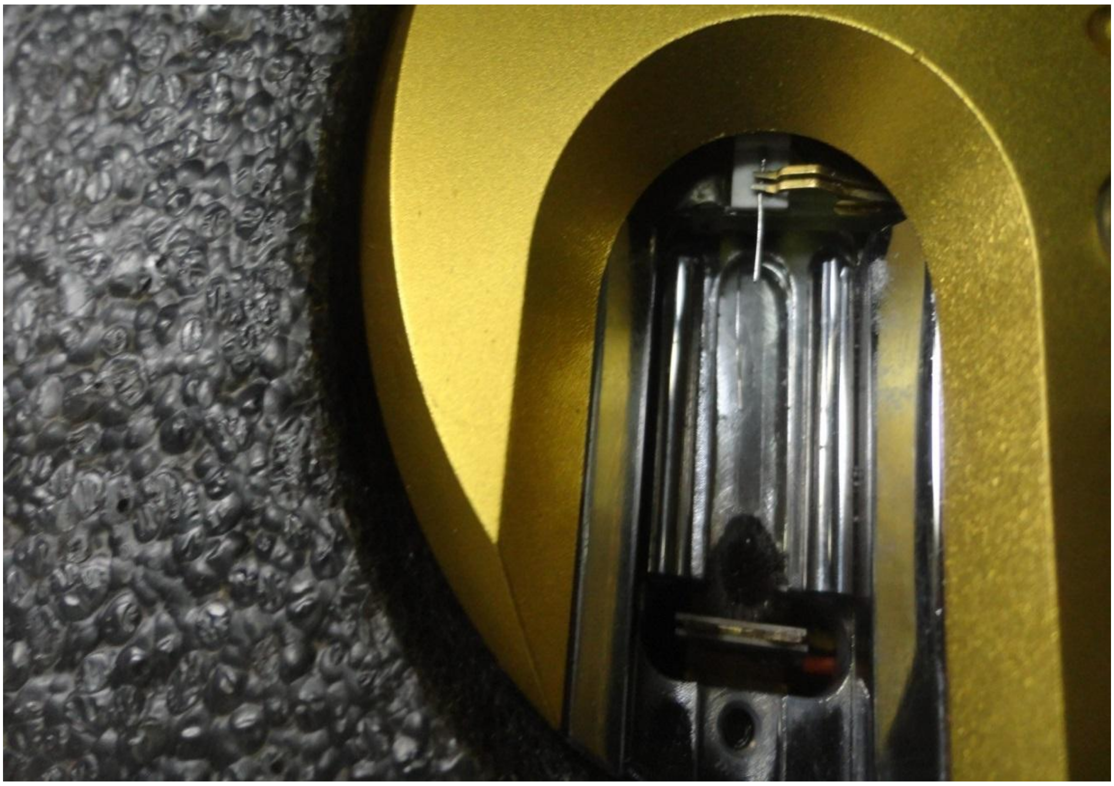

Figure (3.9): show a small sharp platinum tip used in easy Scan device

The tip is scanned over the sample by keeping the current between the tip and sample constant by a feedback loop (constant current mode) the distance between the tip and sample surface is also kept constant and the tip follows the structure of the sample surface. Movement of the tip is recorded during scanning and the “land scape” of the atomic surface can be simultaneously drawn on computer screen line by line show in figure (3.11). That by using specialized software and connections made for this device.

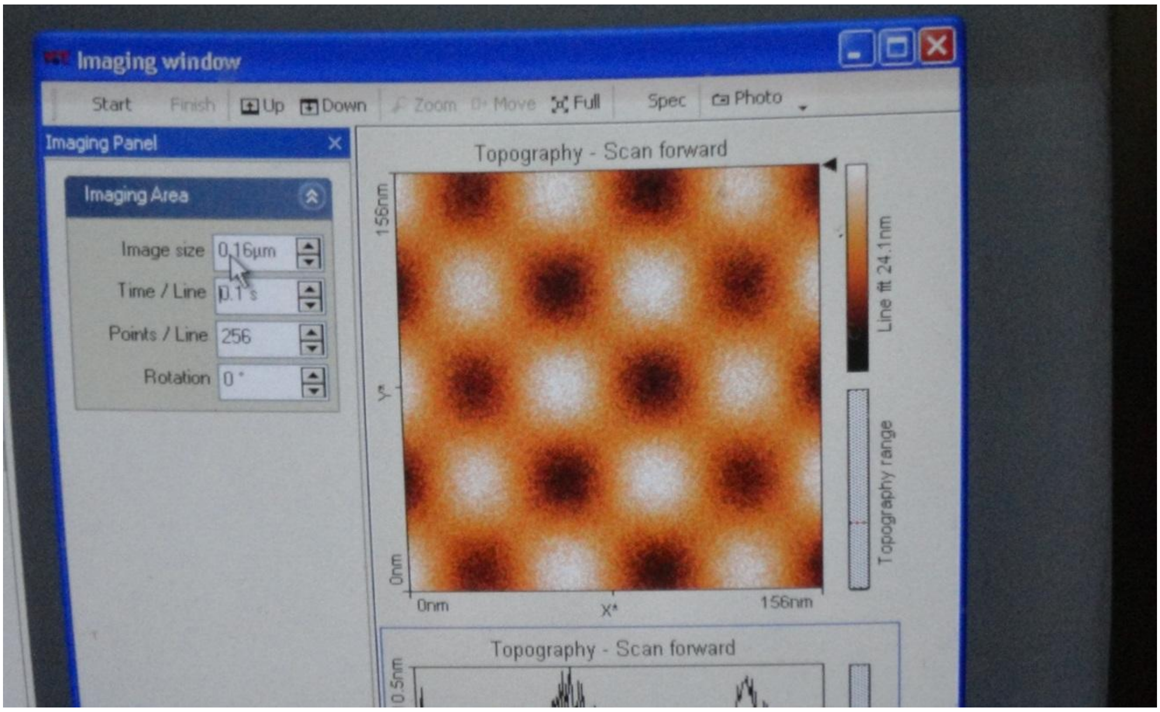

Figure (3.10) show the picture of atoms on computer screen

III. Ultra sonic:

Ultrasonic is an oscillating sound pressure wave with a frequency greater than the upper limit of the human hearing range. Ultrasound is thus not separated from 'normal' (audible) sound based on differences in physical properties, only the fact that humans cannot hear it. Although this limit varies from person to person, it is approximately 20 kilohertz (20,000 hertz) in healthy in Figure (3.12). . Ultrasonic devices operate with frequencies from 20 kHz up to several gigahertzes.

Ultrasonic is used in many different fields. Ultrasonic devices are used to detect objects and measure distances. Ultrasonic imaging (sonography) is used in both veterinary medicine and human medicine. In the nondestructive testing of products and structures, ultrasound is used to detect invisible flaws. Industrially, ultrasonic is used for cleaning and for mixing, and to accelerate chemical processes. Organisms such as bats and porpoises use ultrasound for locating prey and obstacles.

Figure (3.12): show ultrasonic devise (Michael, 2014)