

TREATMENT AND REUSE OF MARBLE FACTORY WASTE



By

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APPROVAL CERTIFICATE

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DEDICATION

We dedicate this thesis to our beloved parents and respected teachers who worked hard and made efforts to make our academic career a successful one and to all of our class fellows for their support. Thank you very much.

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Praise be to Almighty ALLAH who has blessed us with uncountable and unlimited blessings in our life

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ACCRONYMS:

| | |
|---------|--|
| ASTM | American Society For Testing And Materials |
| GDP | Gross Domestic Product |
| IEI | Industrial Estate Of Islamabad |
| IESE | Institute Of Environmental Sciences And Engineering (NUST) |
| L | Liters |
| mg | Milligrams |
| mL | milliliters |
| NICE | NUST Institute Of Civil And Environmental Engineering |
| NTU | Nephelometric Turbidity Units |
| Pak-EPA | Pakistan Environmental Protection Agency |
| rpm | Revolutions Per Minute |
| UTM | Universal Testing Machine |
| XRF | X-Ray Fluorescent Test |

ABSTRACT

Granite and marble processing industries generate excessive amounts of wastes mainly in the form of powder and wastewater during cutting and polishing processes, which pollute and damage the environment. Therefore, this work aims to characterize and evaluate the possibilities of using the granite and marble wastes, generated by the process industries. The waste water with suspended marble and granite particles is normally dumped in open drains where they cause scaling of pipes or dumped in open spaces where they affect natural strata significantly. The foremost objective of the study is to protect the environment through managing the liquid and semisolid slurry waste in sound and economically feasible manner.

The first purpose of the study was to look into ways of incorporating marble and granite waste from the marble processing industry for cladding, ornamental and decorative purposes. The compression and flexure response for different formulations of mortar incorporated with marble and granite waste was investigated and the strength achieved for the mix formulations containing granite was observed to be higher than that containing marble.

Secondly, reuse of marble waste water was made possible using treatment techniques like solids settling (cleaner production approach). The optimum dosage of sludge carrying alum in Granite and marble wastewater was investigated through turbidity tests. For same amount of marble and granite waste water, the sludge amount consumed is double for granite wastewater in comparison to the former.

**IN ORDER TO SAVE THE PLANET AND
LEAVE A LEGACY FOR OUR CHILDREN,
WE ALL NEED TO ENSURE THAT
EVERYTHING WE DO IS SUSTAINABLE, BE
IT AT WORK OR HOME.**

CHAPTER 1: INTRODUCTION

Recycling waste as useful material is a very important environmental management tool for achieving sustainable development. On the other hand, recycling waste without properly based scientific research and development can result in environmental problems greater than the waste itself. Granite and marble process industry generates a large amount of wastes mainly in the form of powder during sawing and polishing processes, which pollute and damage the environment. Therefore, this work aims to characterize and evaluate the possibilities of using the granite and marble sawing wastes, generated by the process industries.

Marble and Granite belong to the category of building stones widely known as Dimension Stone. These are natural stones which can be shaped in form of blocks, slabs, tiles, etc. and are mostly used for monumental and decorative purposes since antiquity. Various civilizations have used dimension stone in many ancient buildings and monuments that have survived to the present day. Stone has played a significant role in human endeavors since earliest recorded history and its use has evolved since ancient time.

The word Marble comes from Greek word “Mamaros” which means shining stone. Marble is a non-foliated, Granular Metamorphic Rock that is formed by the Metamorphism of Limestone and Dolostone. Marble is a Calcium Carbonate (CaCO_3). The term marble is also applied to Serpentine rocks that can be polished to high shine. **Granite** is a common type of intrusive, felsic, igneous rock which is granular and phaneritic in texture. This rock consists mainly of quartz, mica, and feldspar.

1.1. MARBLE DEPOSITS IN PAKISTAN

Almost all provinces in Pakistan have ornamental stone deposits in wide range of colours, shades and patterns. Initial estimates indicate **166 billion tones** of marble reserves across Pakistan (Korai 2012 TDAP) ^[1]. Pakistan has enormous reserves of marble, re-crystallized lime stone, fossil ferrous limestone, dolomite and granite.

Table 1: Marble and granite reserves across Pakistan [1]

| [1] | MARBLE | GRANITE |
|----------------------|---|--|
| RESERVES | Initial estimate are 166billion tones. | Not specifically measured, however more than 1000 billion tons of granite reserves are generally estimated. |
| MAJOR COLOURS | White, Black, Green, Pink, Grey, Brown and Yellow | Black, Pink, Grey, Green, Gold & Yellow and Red |
| LOCATION | Mohmand Agency, Chitral, Buner, Swat, Parachinar, Gilgit, Hunza, Swabi, Bajour, Mardan, Wazirstan, Azad Kashmir, Lasbela, Chagai & Khuzdar. | Gilgit, Dir, Chital, Swabi, Kohistan, Nagarparker, Chagai, Mansehra, Malakand & Swat. Nagarparkar (Sindh) and Mansehra (Khyber Pukhtoonkhwa) are only known sources of workable Granite in the country. Geology evidence shows Gilgit Region holds great promise of the superior quality deposits. |

1.2. PAKISTAN MARBLE INDUSTRY:

Despite that marble and granite have been mined for centuries and mass production was introduced in the 20th century, the marble industry in Pakistan started to develop in late 1960's without any quality consideration. At that time most of the processing equipments were made locally at Gujranwala and Lahore. In

mid 1970's, few plants were imported from Italy which were of 2nd generation plants. Only in late 1970's, few large companies and wealthy private businessmen imported modern machineries and focused on higher quality products. Since 1990, mining and quarrying of marble has consistently contributed by 0.5 percent to Pakistan's GDP. The industry also employs around 40,000 workers. There are around 17 active quarries and 1600 processing plants. Around 150 to 160 processing plants are equipped with appropriate machineries. By producing more than 900,000 tons of marble, Pakistan only contributes by 1 percent to world total production. In 2010, Pakistan's marble and granite exports reached \$60 million (Rassin. 2012)^[2].

Table 2: Basic information about Pakistan's marble industry

| INDICATORS | PAKISTAN |
|--|----------|
| Total production (tons) | 950,000 |
| Average annual growth rate (percent) | 20 |
| Number of firms | 1600 |
| Export value in 2011 (million US\$) | 60 |
| Number of workers involved | 40,000 |
| Total value of marble industry in 2011 (million US\$) | 650 |
| Share in GDP (percent) | 0.5 |

1.2.1. Importers of Pakistan's stones:

Marble and granite are produced in more than 40 countries in the world. Italy, Turkey, Spain, India and China are the top five dominant countries in terms of marble production. These countries control over half of the world market – only Italy produces over 17 percent of world marble. A major part of production is consumed locally by producing countries, and only a small percentage of total

production is exported. This fact indicates that local supply of marble remains less costly, while the transportation cost increases the price of exported marble products. The export during the last four years has significantly increased in marble and granite sector, which is estimated at **US \$ 35,814,000** (Khan, 2012) ^[4]. Pakistan's Marble trade is growing as China is importing the raw marble blocks from Pakistan. According to statistics issued by Ministry of Commerce, China imported **70,790 metric ton** of Pakistani marble from July to March 2010-11. This is the huge quantity of marble export to china. The most part of this export was raw material in the shape of raw and squared blocks which is very low priced in Pakistan. Among other top importers of Pakistani natural stone from July to March 2010-11 are (Millat, 2011) ^[3]:

Table 3: List of major Importers of Pakistan's marble

| Importer | Quantity of Marble (metric ton) |
|---------------------|--|
| UAE | 2,994 |
| Saudi Arabia | 2,515 |
| Italy | 2,414 |

The major marble/granite deposits in KPK are located in Buner, Mardan, Swat, Mohmand and Bajaur area. Buner that used to have mountains covered with naturally green trees, herbs and shrubs is now presenting a very devastating picture. Buner is a hub of marble/granite mining and processing with at least 400 mines, almost all extracting the natural endowment through unprecedented blasting that leave;

Table 4: Some important negative impacts of marble quarry

| S.NO. | Negative Impacts |
|-------|---|
| 1. | Harmful effects on the environment |
| 2. | Destroy scenic landscaping of the area |
| 3. | Destroy trees, herbs and shrubs on the hills where mining activity is carried out |
| 4. | Damaged natural resource almost 75% loss on mines |

There is also a huge processing industry with 350 processing units spread in the entire Buner valley. These processing units further aggravate the situation by spilling waste in the water ways and in the air. Environmental degradation and processing efficiency is threatened with;

- ✘ Flow of slurry in the water channels that contaminate the entire water flow in the valley.
- ✘ Solid waste of the processing units is disposed off on the road side in the vicinity of the processing unit.

1.3. CHEMICAL COMPOSITION

1.3.1. Marble:

Chemically, Marble is composed predominantly of calcite, dolomite or serpentine minerals. The chemical composition of marble, by weight percentage is given as (Pascal, B. 2011) ^[5]:

Table 5: Chemical composition of marble

| S.N. | MINERALS | WEIGHT PERCENTAGE |
|------|--------------------------------------|-----------------------------|
| 1 | Lime (CaO) | 28-32% |
| 2 | Silica (SiO ₂) | 3-30% (varies with variety) |
| 3 | MgO | 20 to 25% |
| 4 | FeO + Fe ₂ O ₃ | 1-3% |
| 5 | Loss On Ignition (LOI) | 20-45% |

1.3.2. Granite:

The chemical composition of granite, by weight percentage is given as (Harvey et al. 1997)^[6]:

Table 6: Chemical composition of granite

| S.N | MINERALS | WEIGHT PERCENTAGE |
|-----|--|-------------------|
| 1 | SiO ₂ (silica) | 72.04% |
| 2 | Al ₂ O ₃ (alumina) | 14.42% |
| 3 | K ₂ O | 4.12% |
| 4 | Na ₂ O | 3.69% |
| 5 | CaO | 1.82% |
| 6 | FeO | 1.68% |
| 7 | Fe ₂ O ₃ | 1.22% |
| 8 | MgO | 0.71% |
| 9 | TiO ₂ | 0.30% |
| 10 | P ₂ O ₅ | 0.12% |
| 11 | MnO | 0.05% |

The main objective of waste management system is to maximize economic benefits and at the same time protection of the environment. Granite and marble process industry generates a large amount of wastes mainly in the form of powder

during sawing and polishing processes, which pollute and damage the environment. Therefore, this work aims to characterize and evaluate the possibilities of using the granite and marble sawing wastes, generated by the process industries. The figure below shows the stages of marble/granite processing and subsequent environmental degradations caused by them:

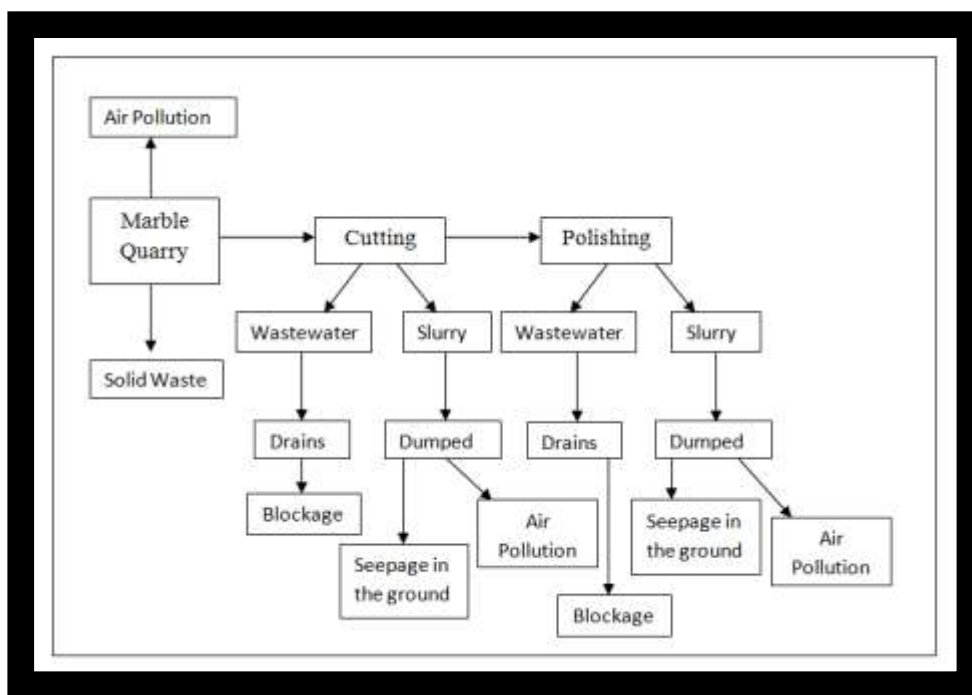


Figure 1: Schematic diagram of stages and impacts of marble processing

1.4. OBJECTIVES OF STUDY

The primary objective of the project is to grasp the environmental degradation through introduction of model cleaner production practices and facilitation in development of pollution control systems for marble/granite and stone crushing units by utilization of marble/granite and granite slurry / powder into marketable value added marble/granite products notably bricks / blocks. The objectives of our study were same and were fulfilled using following options:

1. Re-use of wastewater using solid settling approach

2. Value addition to the slurry waste by making tiles for ornamental purposes

The secondary objectives of the project are to:

- To control dust and waste water pollution generated during stone crushing and marble/granite cutting processes, respectively.
- To recommend cleaner production practices to minimize the negative impact of marble/granite slurry on environment so as to minimize hazards emanating from slurry.

CHAPTER 2: REVIEW OF LITERATURE

2.1. MARBLE & GRANITE PROCESSING AND WASTE GENERATION:

Marble processing usually involves three steps quarry, cutting and polishing. References estimate that 20 to 25% of the marble/granite produced results in powder in the form of slurry, as for each marble or granite slab of 20mm produced; 5 mm is crushed into powder during the cutting process (S. El Hagggar, 2007) ^[7]. These processes are discussed in detail below:

2.1.1. Quarry:

The first stage of the production is the identification of a quarry location in finding an optimal deposit of material with desirable color, pattern, and composition. Samples are then obtained by boring into the earth to take core samples with expensive diamond-tipped drill bits. These samples are then tested to determine if the stone is suitable for use as dimensional building stone. Once this is done, the owner applies for a concession for the quarry area. After the concession is purchased, the next step is to apply for all of the correct licenses from the local, state and federal government. Clearing the way to reach the best material allows a quarry to reach its full potential. Each quarry presents a unique challenge for the extraction of blocks. There may be months of removing overburden, or dirt, before the stone is accessible.



Figure 2: Drilling out marble from quarry

Drilling is the second process. The process starts by taking down a “bench wall,” a large dimensional chunk of rock that is then cut into smaller blocks which will eventually be sent to the factory for processing. The bench walls are cut using a combination of diamond wire cables, drills and even high temperature torches that will melt the stone. Dirt is pushed up against the base of the wall to cushion the fall, and small dynamite charges jar the wall loose to bring it down to a horizontal position. The blocks can then be drilled from the bench wall. There are main two type of the driller is used.

- (1) Pneumatic steel driller
- (2) Pneumatic down-the –hole driller.

Blocks of a given type of stone usually have a fairly uniform size, due to the size of the processing equipment used. Granite blocks usually weigh between 38-42,000 pounds, while lighter marble and travertine blocks weigh between 15-25,000 pounds.

2.1.2. Cutting:

Once blocks of stone reach the processing facility, they are cut down into smaller more manageable pieces. For tile, this means cutting the stone into billets before polishing. For slab materials, this typically means a trip to the gang saw. A gang saw works just like a giant bread slicer, with many adjustable blades that allow for the thickness of the slabs to be adjusted. The gang saw can cut the entire block of stone into slabs at one time. More delicate materials may require the use of a diamond wire saw to gently cut one slab at a time with fewer traumas to the stone. The most recent technology involves multi-wire gang saws which have the potential speed production on exotic materials exponentially. Water is showered on blades while stone blocks are cut into sheets of varying thickness to cool the blades and absorbs the dust produced during the cutting operation. The amount of wastewater from this operation is very large. It is not recycled as the water so highly alkaline that, if re-used, it can dim the slabs to be polished. In large factories, where the blocks are cut into slabs, the cooling water is stored in pits until the suspended particles settle (sedimentation tanks), then the slurry is collected in trucks and disposed of on the ground and left to dry. This water carries large amounts of stone powder. Eventually, the sludge dries in the sun and its particles become airborne. This causes air pollution problems for the surrounding area. Another solid waste generated by the marble and granite units is the cutting waste which results from cutting slabs into the required dimensions.



Figure 3: Cutting of large tons into small thickness slabs using large blades and spraying water



Figure 4: Cutting/sawing into tiles and slabs with spraying water

2.1.3. Polishing:

This is the last process of marble processing. After slabs are cut on the gang saw, they are moved one at a time to the polishing line, where they are laid horizontally on a large conveyer type line called a polishing line. There, they pass under polishing heads which begin with very coarse diamond abrasives, and then move to finer and finer grit abrasives, just like sanding wood. Travertine slabs and some marbles, will receive a cementacious or epoxy compound to fill the natural voids of the stone.

Part of the way through this line, slabs of granite, marble, and onyx will receive a coating of a resin treatment which will fill in any pits or micro fissures which are inherent to the stone in order to make the final surface easier to clean. Most of the excess resin is removed by further polishing, with only 1% remaining on the finished surface. For materials where the final surface is to be honed, the process will stop with a lower grit abrasive than materials with a polished surface. The polishing operation is fully automated with the use of powdered abrasives that keep on scrubbing the surface of the marble until it becomes smooth and shiny. Water showers are essential to prevent overheating of the blades.



Figure 5: Polishing the marble surface to add shine using water

2.2. ENVIRONMENTAL IMPACTS:

Marble and granite industry is one of the most environmentally unfriendly industries. Cutting the stones produces heat, slurry, rock fragments, and dust. Although marble waste, in general, includes non-radioactive by-products, and thus it does not induce climate changes, it does destroy plant life. Marble waste cannot be considered inert; the fines are alkaline (appendix 2) materials producing high pH wastes.

The fine particles can cause more pollution than other forms of marble waste unless stored properly in sedimentation tanks, and further utilized. The fine particles can be easily dispersed after losing humidity, under some atmospheric conditions, such as wind and rain. The white dust particles usually contain CaCO_3 and thus can cause visual pollution. Clay and soils have a high cation exchange

capacity and can absorb high proportion of heavy metals and cations, such as Ca, Mg, K and Na; yet soils are not as effective as marble and granite fine particles in holding pollutants like Cl. The waste in the water does not completely sink to the ground, and much of it remains on the surface. As the water on the surface evaporates, the liquid wastes solidify. Meanwhile, relatively wet marble waste, which is subjected to rain and snow, will be carried with seepage down into the ground over time (Celik et al. 2008) ^[8].

The marble slurry could lead in the long run to water clogging of the soil, to increasing soil alkalinity, and to disruption of photosynthesis and transpiration. The net effect is a reduction of soil fertility and plant productivity. Factory waste dumped in open spaces can result in enhancing airborne pollution.

According to a report of Pakistan Environmental Protection Agency (Pak-EPA), 31 marble factories are operating in the industrial estate of Islamabad (IEI). Their manufacturing processes included cutting and polishing of marble slabs and tiles.

Initially, these units were using dry cutting process but due to regulatory measures, most of them switched over to wet cutting process. “Significant quantity of water is used to cool blades and catch the dust formed during the cutting process. Water is showered on blades while marble blocks are cut into sheets of various thicknesses.

Resultantly, the water cools the blades and absorbs marble dust produced during the process,”

The wastewater is routed to a series of settling tanks, in which marble dust settles down partially and water is recycled. Effluents discharge into channels: The

excess water containing sludge is discharged into effluent channels of the industrial estate, which later passes throughout the capital. This water carries large amounts of marble powder, which gradually settles in bottom of the drain channels. After mixing with other chemicals present in channels, it may spoil the underground water of adjacent areas.

Moreover, the marble sludge in the settling tanks is periodically removed and dumped in the vicinity, eventually, the sludge after getting dried causes air pollution problems for the inhabitants of the surrounding areas. Disposal of sludge is a major environmental problem. Another solid waste generated by the marble units is the cutting waste.

2.2.1. Threat to flora and fauna:

These marble particulates destroy plants by covering their leaves and reducing their access to sunlight, ultimately trees die and vegetation in the area reduces. Studies show that these particles are also highly dangerous to marine and animal life in the area.

2.2.2. Impacts on human health:

The workers and residents living in adjacent areas are prone to a disease called silicosis,” In silicosis, marble particles damage cells of respiratory system and people feel difficulty in breathing. There is no treatment of this disease and it leads to respiratory system failure, which could result in death of the patient. There is no cure to this disease and only avoidance of dust can improve the patients (DailyTimes 2008) ^[9].

2.3. DIFFERENT OPTIONS AVAILABLE FOR THE USE OF MARBLE/GRANITE SLURRY FROM LITERATURE

2.3.1. As filler in rubber, plastics, fiber glass and paints.

i. White and coloured tiles

Process:

- Sieving
- Dewatering
- Mixing with white cement / Lime
- Die forming in hydraulic press.

ii. Decoration pieces / bricks / blocks.

Washed graded slurry mixed with white cement can be casted into different shapes.

Binders other than white cement such as Lime, PCA (white Glue) or fiber glass, (unsaturated polyester resins) can also be used. Slurry needs to be washed, graded and dewatered. For resin bonding, it must be completely dried. For conversion into bricks / blocks mixing of Silica / Cement / Lime can be feasible and economical. If innovative polishing techniques are introduced the bricks / blocks can be marketed at higher price to affluent class.

The book **‘Environment Friendly Indian Building Material Technologies for Cost Effective Housing’** by the author, President, Society for Excellence in Habitat Development, Environment Protection and Employment Generation (SHEE) includes variety of production technologies of building

materials as developed and practiced in India. The technologies utilize locally available raw materials, wastes and by-products from industry, agriculture and natural fibres.

2.3.2. Marble/granite slurry bricks:

Marble/granite slurry is being used to make marble/granite slurry bricks. This technology is in practice from a long time and the materials required, the process and its benefits are given below:

Use: For walling as an alternative to conventional clay bricks

Size of Product: 230x115x75mm

Properties of Product:

- Compressive strength 93kg/cm²
- Water absorption 14%
- Volume of brick 1687.5cm³
- Color White/Grey

Raw Material:

- Marble/granite Slurry 83%
- Cement 7%
- Sand 10%

**Table 7: Machinery and equipment requirements for various options
being used in India for marble/granite powder**

| | |
|---|---|
| OPTION 1: Vibro Press (Sakar) | OPTION 2: Bi-Directional Press (Vibro Hydraulic Compaction Type-As 1818) |
| Production Capacity: | |
| 1.8 million bricks per annum (2 shifts of 8 hours each) | 3 million bricks per annum (2 shifts of 8 hours each) |
| Equipment/Machinery: | |
| <ul style="list-style-type: none"> • Brick making machine (Sakar), • Pan mixer • Extra Moulds | <ul style="list-style-type: none"> • Bi-Directional Vibro Press (AS-1818) • Pan mixer • Belt conveyor • Box feeder • Pallets |
| Land Requirement: | |
| <ul style="list-style-type: none"> • Open are: 3000m² • Covered area: 150m² | <ul style="list-style-type: none"> • Open area: 3000m² • Covered area: 200m² |
| Power Requirements: | |
| <ul style="list-style-type: none"> • KW: 12.75 • Three phase • Voltage: 440V, 50Htz | <ul style="list-style-type: none"> • KW: 22 • Three phase • Voltage: 440V, 50Htz |

2.4. BENEFITS OF THE PROJECT

The perception of the waste in the sustainable industrial development has changed, and it is now considered as “gold”. Waste of one industry can be used as an input in another industry and at a much cheaper cost. Moreover, the new mode of industrial activity has been transformed into a more integrated one called an

industrial Ecosystem. In such system, the consumption of energy and material is optimized, waste generation is minimized, and effluent of one process serves as the raw material for another process. Transfer to clean technology, mechanisms for recycling, good housekeeping inside industry, relying on local resources and the creation of new environment friendly industries are the only way by which Pakistani industry will stay competitive in world market in future.

The project, “Introducing Cleaner Production Practices and Controlling Environmental Degradation in Marble/granite Industry to ensure Sustainable Development” by (Siddiqui 2012) ^[12] have discussed options for the above purpose to get benefits as given below:

- The cleaner production practice will reduce the slurry waste, which will be utilized for further beneficial utility products and will eliminate waste pollution.
- It will help and guide marble/granite factories in implementation of Pakistan Environmental Protection Act 1997.
- The system will help Marble/granite and Stone Crushing units to comply with National Environmental Quality Standards.
- In marble/granite units, the water after settling of slurry will be recycled thus the consumption of water will be kept at minimum level and the risk of damage to agriculture and aquatic life will thus be reduced.
- There will be less concentration of pollutants in the main drains where these units are located.

- The project would encourage and facilitate the marble/granite sector towards handling, compliance issues and meeting the international quality standards and best manufacturing practices.
- Control of dust emission from stone crushers will drastically reduce the risk of negative effects on health and wellbeing of human and on vegetation in the vicinity.
- The re-use of properly treated water will increase the useable life of the cutting blades and will also consume less water.
- The proposed system will yield both economic and environmental benefits.

CHAPTER 3: MATERIALS AND METHODS

3.1. KOHSAR INDUSTRIES:

For this study, selected industry was Kohsar industries that was established in 1983, and now is one of the leading marble factories of Pakistan. This is located in sector I-9/3 Islamabad. The owner of Kohsar Industries approached IESE to help him with the management of the factory waste. Owner of unit, Mr. Farrukh Munir is also an active member of Pakistan Chamber of Commerce & Industry, Islamabad. This industry produces export quality marble and granite processed on Italian machines. Main process done here, involve marble and granite cutting and polishing. According to the data collected from the factory, they are processing 20 to 25 tons of marble and granite per day with consumption of approximately 100 m³ of water. Daily operation timings of the factory are 8 hours.

3.2. SAMPLING:

Composite sampling was done from the factory to pursue with the objectives of the study. Kohsar industry has three tanks each of (20 X 10 X 6) feet, where they allow water to stand for sometimes for gravity sedimentation and then dump settle solids in open spaces and drain the supernatant in the sewerage lines. The 7 days settled solid from these tanks was collected for testing and liquid samples were collected from working area. (Appendix 3)



Figure 6: Industry selected to pursue with thesis objectives



Figure 7: Water tanks used to store wastewater on site



Figure 8: Liquid sample collection after step 2 cutting



Figure 9: Wastewater going into open drains without any treatment

3.3. WATER RE-USE APPROACH:

As already mentioned in chapter 2, the chemical composition of the stone is alkaline. The water wasted from the processing in the factory contains waste 25% of the original stone (Rania et al. 2011) ^[10] that increase the pH of the water. This water cannot be used as it is because it is highly alkaline.

Raw water collected from the Kohsar industry had turbidity greater than 3000NTU. This is very high turbidity representing high levels of TSS in the raw wastewater. The target was to lower the turbidity to 50 NTU to make it re-useable by the factory themselves for processing of stones. Cleaner production practice is introduced for enhancing the settle-ability of suspended solids. Sludge from the water treatment plant Rawal Lake was used to treat this water. The sludge contained the alum dosage which was different for the different months of the year. The alum dosage from October to January was 20 to 50 mg/l and from February to September it was 20 to 120 mg/l. The tests were conducted from October to January; therefore the results that will be presented in this study will be based on the alum dosage 20 to 50 mg/l.

Settling velocity of the particles depends on the particle size. Alum is coagulant that enhances the size of the particles by forming flocculates. Enhancing the settle-ability reduces the time to reach the desired results. This fact was proved by the study carried out in wastewater lab IESE.

3.3.1. Equipment & material

Two batches of the tests were conducted. First one was to find settling of suspended solids in marble and granite wastewater without the alum sludge dosage.

Second one was to find the optimum ratio of sludge to sample that must be used to get desired turbidity in less time. The equipment used is:

1. 250 ml beakers
2. Pipette
3. Stirrer
4. Quartz cell
5. Turbidity meter
6. Tape and marker

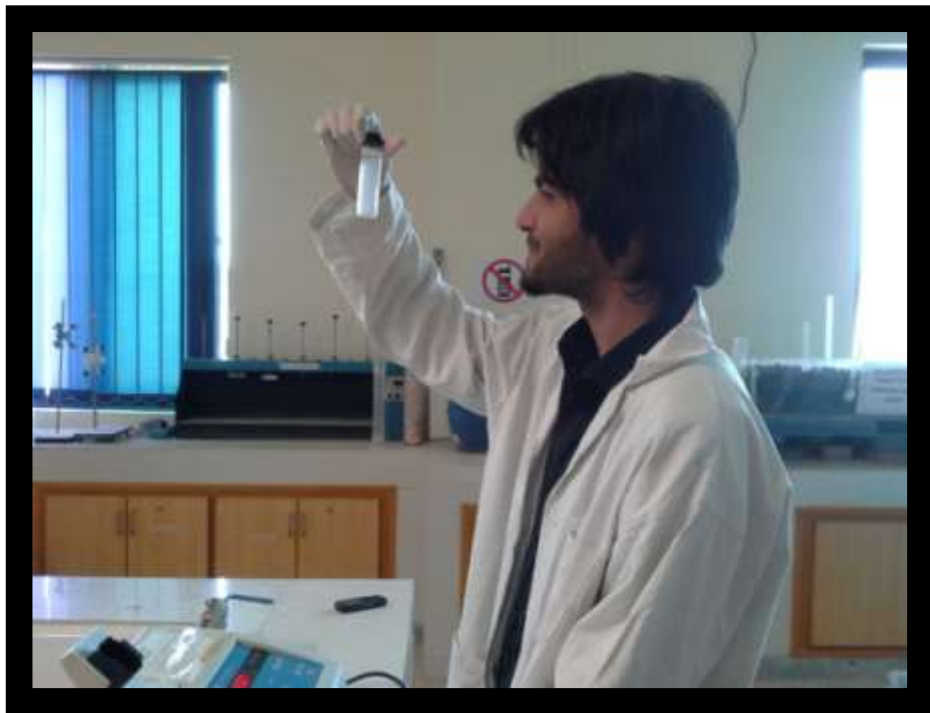


Figure 10: Quartz cell used for turbidity reading in turbidimeter



Figure 11: Turbidity meter for measuring the turbidity of effluent

3.3.2. Experimentation:

The first batch methodology was conducted to find the settling over time without adding Alum sludge and is shown in the flow chart below:

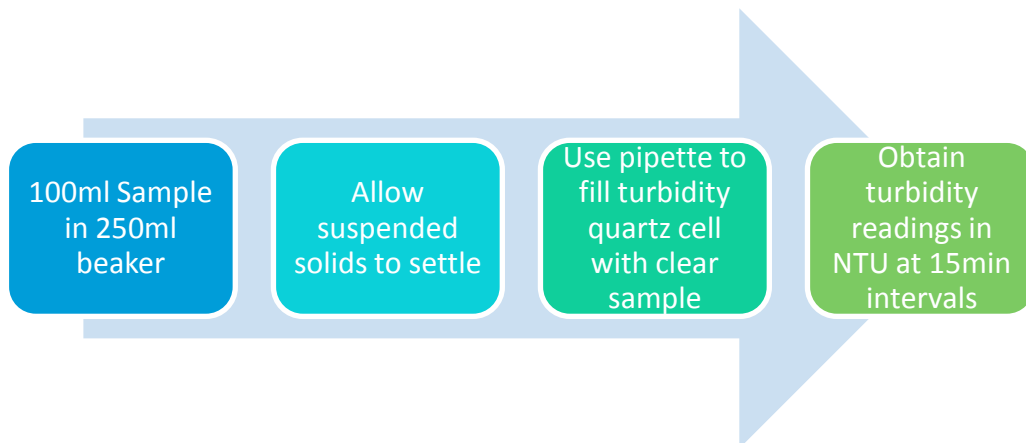


Figure 12: Flowchart showing turbidity testing over time without alum sludge

Second batch methodology to find optimum dosage is shown in the flowchart below:

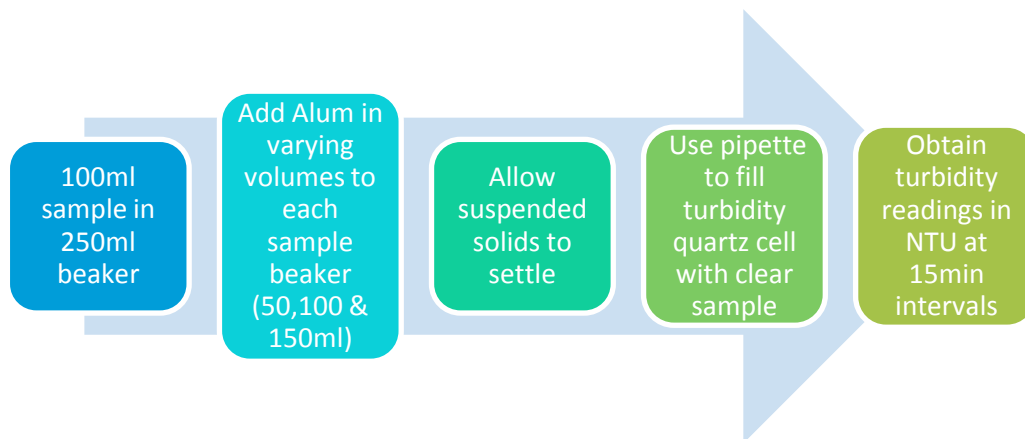


Figure 13: Flowchart showing turbidity testing over time with alum sludge



Figure 14: Experimental setup for turbidity removal study

The results obtained from both the studies are analyzed and compared by graphing the relationship between decreases in turbidity with time. The results expected are to show that settling time has been reduced by adding alum sludge from Rawal Lake water treatment plant and secondly to find optimum alum sludge dosage in the wastewater.

3.4. SLURRY RE-USE:

The composite samples collected from the Kohsar Industries were firstly dried in backyard of IESE NUST. The important factor was to determine the chemical composition of the dried sample for further testing. From literature^[2] it was clear that the calcium content in marble is higher where in case of granite it was silicon dominating. Silicon content in granite makes it naturally hard.

Although marble and granite powder are used for variety of purposes already, but in this study tests were conducted to look into ways of incorporating marble and granite waste from the processing industry for cladding, ornamental and decorative purposes. The compression and flexure response for different formulations of mortar incorporated with marble and granite waste was investigated and the strength achieved for the mix formulations containing granite was observed to be higher than that containing marble.



Figure 15: Sample drying in open air at backyard of IESE-NUST



Figure 16: Dried marble powder used for further making bricks and tiles

3.4.1. Moisture content (mc):

First of all moisture content of sample was determined using ASTM 313.

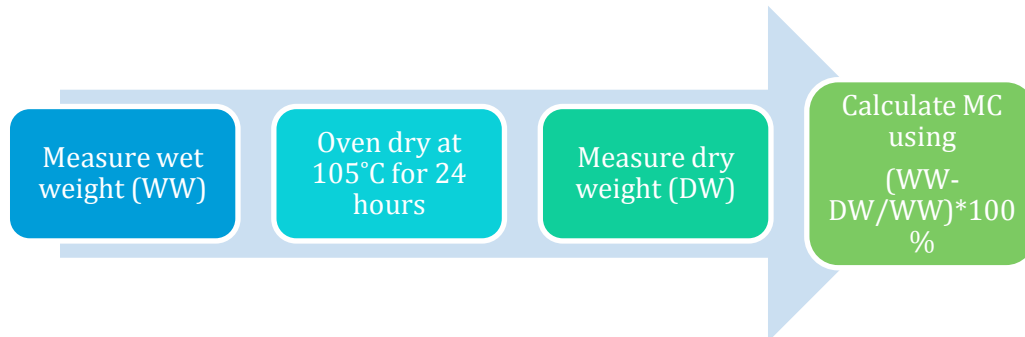


Figure 17: Moisture content flowchart

Average value of three samples was taken and following results were obtained:

- ✓ **Marble Sample = 18.8%**
- ✓ **Granite Sample = 17%**

3.4.2. Chemical composition:

X-Ray Fluorescent method was used to find the composition of marble and granite powder. XRF machine shown in the figure in IESE lab was used. 100 grams of each marble and granite oven dried powder sample was used to find the composition in XRF. The detail results are attached in appendix 2. Summarizing the results, marble contains 98.6% relative mass percentage of calcium. Granite sample contains 61.5% relative mass percentage of silicon. The analysis with oxides represented high relative percentage of calcium oxide in marble sample and silicon dioxide in granite sample.



Figure 18: XRF machine used to find chemical composition of the dried samples

3.4.3. Cladding tiles-compression tests:

3.4.3.1. Compressive strength test:

Indian standards are followed to make the cladding tiles. The ratios according to Indian standard are cement: sand = 1:3 and 1:2. Water requirement for making the paste is calculated according to the same standard i.e. 0.4 of cement. The project study was conducted to partially replace sand with marble and granite powder. Three and two parts of sand were replaced by different percentages of marble and granite powder. These formations were tested for compressive strength

>1500 psi. According to ASTM C648 (appendix 4), these cladding tiles were tested in Universal Testing Machine (ASTM International) ^[11].

3.4.3.2. Initial calculations:

Initially prisms (4X4X16 cm³) with different formulations are casted. For casting little calculation was done shown below:

$$VOLUME\ of\ Prism = 4 \times 4 \times 16 = 256\ cm^3$$

$$Mass\ of\ Prism = volume\ of\ Prism \times density\ of\ concrete$$

$$Mass\ of\ Prism = 256\ cm^3 \times \frac{2400\ gm}{m^3} \times 10^{-3} \frac{m^3}{cm^3} = 614.4\ gm$$

Therefore, one prism will contain total 614 grams of cement + sand +marble/ granite powder. Water requirement was calculated by hit and trial method. For sand replacement with marble, water added was 0.4C+0.3M while for sand replacement with granite, water added was 0.4C+0.27G. The calculated grams for each percent replacement of sand with marble are shown in table below:

Table 8: Prism composition used to make different samples for strength tests

| SAMPLE | WATER | CEMENT | MARBLE | SAND | PERCENT REPLACED |
|------------------|--------------|--------|--------|------|------------------|
| 1 | (0.4C+0.35M) | 1 | 2.7 | 0.3 | 90 |
| Weight gm | 221.75 | 165 | 445 | 49.5 | |
| 2 | (0.4C+0.35M) | 1 | 2.8 | 0.2 | 93.33 |
| Weight gm | 227.7 | 165 | 462 | 33 | |
| 3 | (0.4C+0.3M) | 1 | 2.6 | 0.4 | 86.67 |
| Weight gm | 194.7 | 165 | 429 | 66 | |
| 4 | (0.4C+0.3M) | 1 | 1 | 1 | 50 |

| | | | | | |
|------------------|-------------|-----|-----|-----|----|
| Weight gm | 154 | 220 | 220 | 220 | |
| 5 | (0.4C+0.3M) | 1 | 1.5 | 0.5 | 75 |
| Weight gm | 187 | 220 | 330 | 110 | |
| 6 | (0.4C+0.3M) | 1 | 0.5 | 1.5 | 25 |
| Weight gm | 121 | 220 | 110 | 330 | |

Similar calculations are done for granite samples.



Figure 19: Mould used for making prism

3.4.3.3. Equipment & experimentation:

The equipment used for casting and testing are:

1. Weighing machine
2. Hobart Mixer
3. Universal testing machine (UTM)
4. Moulds (4X4X16 cm³)



Figure 20: Mixer for mixing different composition



Figure 21: Prisms prepared for compression strength tests

First step is to weigh calculated grams of sand, cement, marble and water and add all in to mixer. Following the ASTM Standard E196, turn on Hobart mixer at a slow speed of 52.4 rpm for 30 seconds, stop it for 30 seconds, and turn mixer

on again for 90 seconds at high speed of 125 rpm. Pour the mixture into mould making sure there are no air bubbles. Casted Prisms are taken out after 24 hours and then water cured for testing. According to ASTM C648, the prisms are water cured and tested for 7, 14 and 27 days to check the improvement in the results. The optimum replacement percentages selected will be those giving compressive strength >1500psi in the 7days compressive strength.



Figure 22: Compressive strength test in UTM machine

CHAPTER 4: RESULTS AND DISCUSSION

4.1. TURBIDITY:

The results are analyzed to get the optimum ratio of sludge to sample and to observe the benefit of using alum sludge. The results of test carried in figure 14 clearly represented the optimum sludge dosage to get below 50NTU. The result figure 24 represents turbidity results for marble wastewater with varying sludge concentration added.

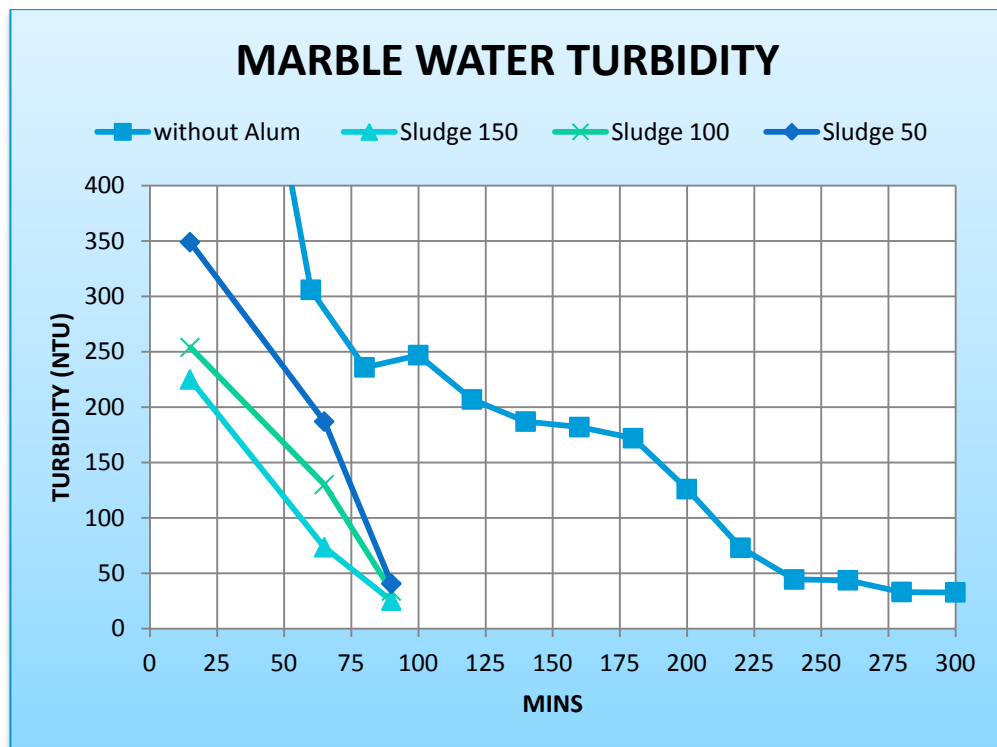


Figure 23: Marble water turbidity at different times for different alum dosage added

It is clearly observed that at 90 minutes all the alum sludge to marble sample ratios give turbidity below 50 NTU. Therefore optimum dosage selected was 0.5:1 which means for treating 1L of marble sludge 0.5 L of alum sludge dosage is required which contains 17.5 mg of alum approx. (concentration of alum

in sludge calculations are for the months October to January). When allowed to settle without alum sludge, the target NTU was obtained at 240 minutes.

The result in figure 25 represents turbidity results for granite wastewater with varying sludge concentration added. It is clearly observed that at 120 minutes the alum sludge to granite sample ratios 1:1 gives turbidity below 50 NTU. Therefore optimum dosage selected was 1:1 which means for treating 1L of granite sludge 1L of alum sludge dosage is required which contains 35 mg of alum approx. (concentration of alum in sludge calculations are for the months October to January). When allowed to settle without alum sludge, the target NTU was obtained at 260 minutes.

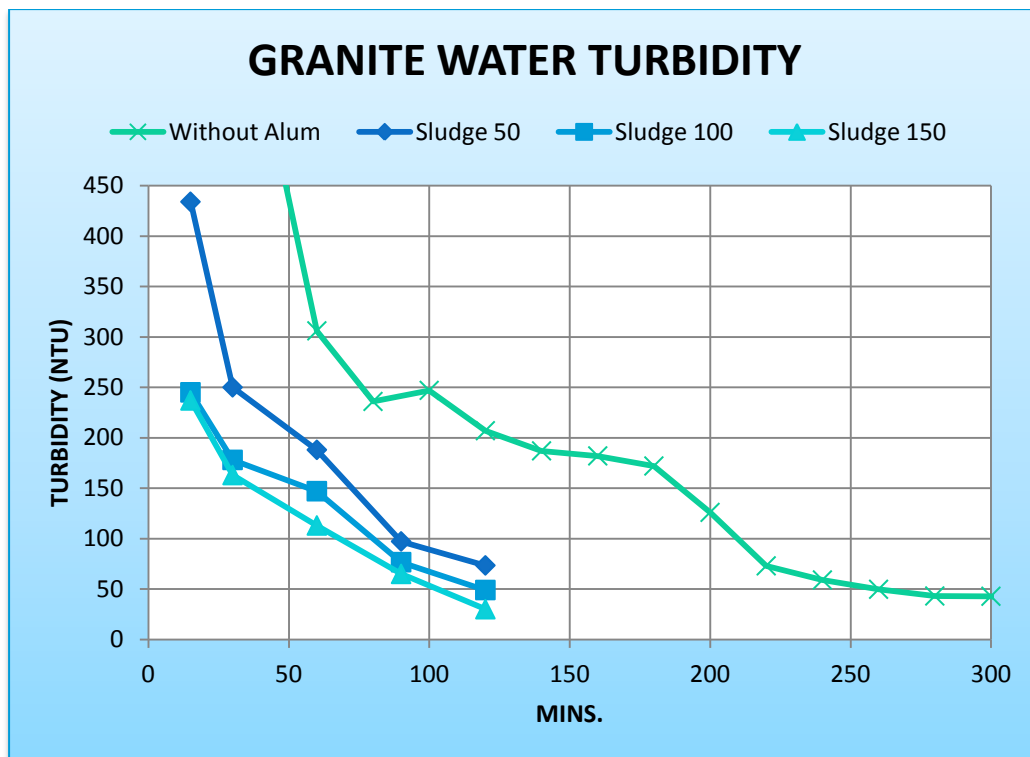


Figure 24: Granite water turbidity at different times for different alum dosage added

4.2. COMPRESSIVE STRENGTH RESULTS:

There is clear trend observed that as sand replacement by marble powder is

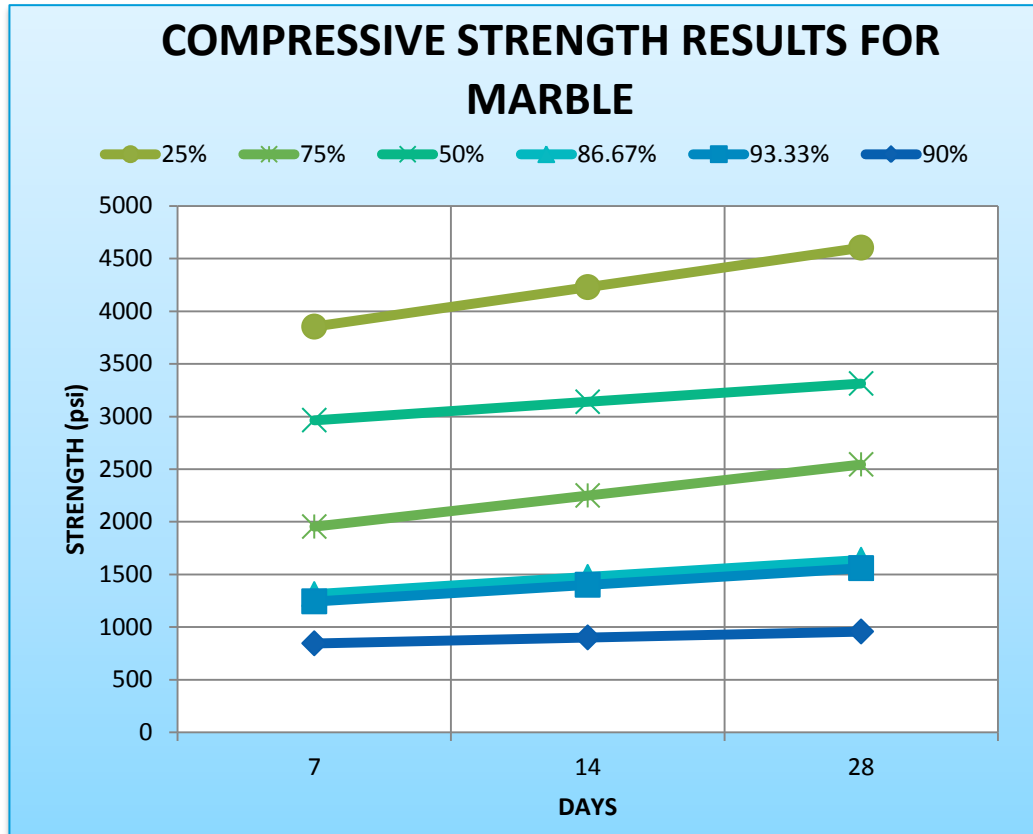


Figure 25: Compressive strength results for marble prisms

increased, strength decreases. The strength results are shown in figure below:

The figure clearly represents that at 75 % replacement of sand with marble powder, strength is >1500 psi and same for 50% and 25%. We want maximum replacement of sand with marble powder therefore; optimum replacement percentage selected is 75% and 50%.

The results shown by marble powder and keeping in view that granite is harder than marble same replacement percentages are used for granite testing and results of compressive strength are shown in figure.

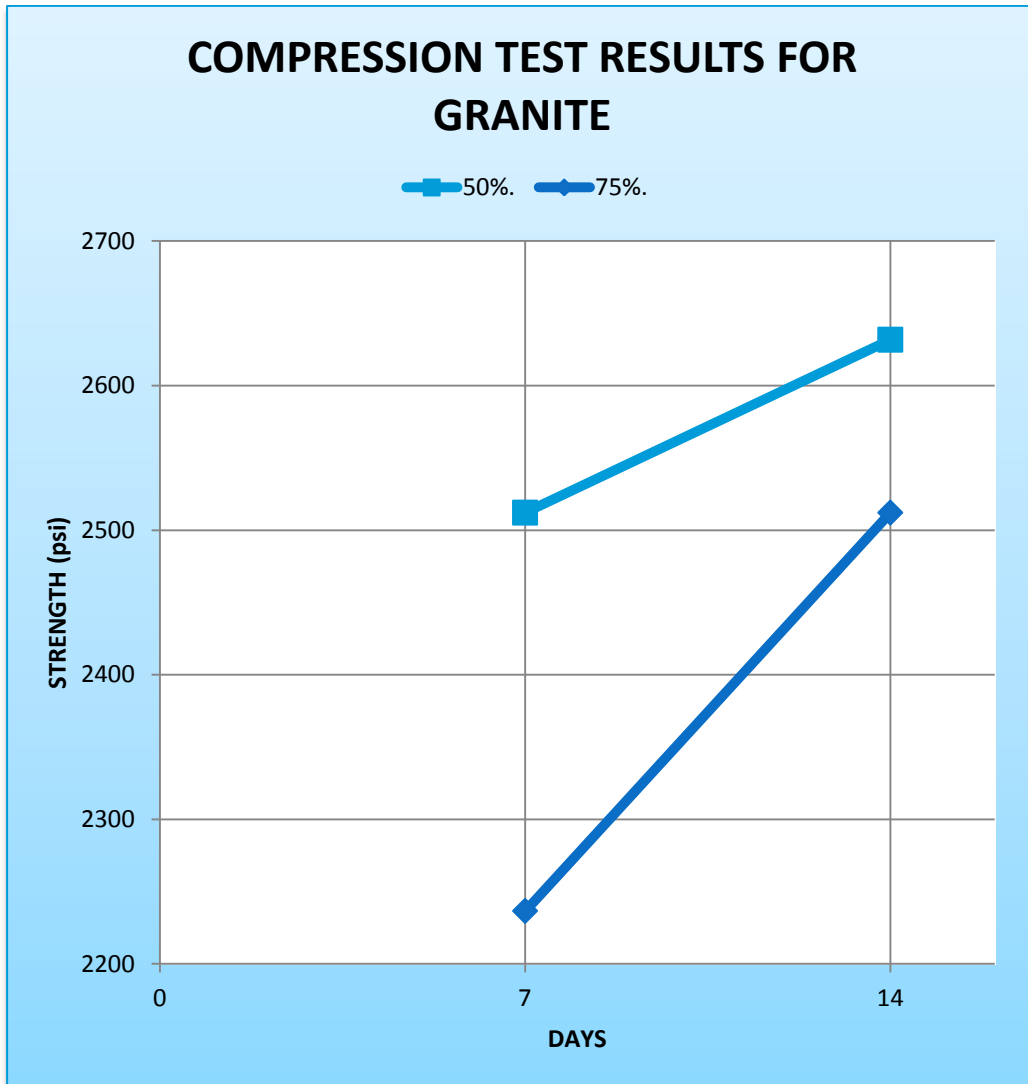


Figure 26: Compressive strength results for granite prisms

4.3. CLADDING TILES USE:

The proposed solution was to use marble/granite powder to make cladding tiles. In building construction, cladding refers to the application of one material over another to provide a weather-proof layer intended to control the infiltration of weather elements. Cladding does not necessarily have to provide a water-proof condition but is instead a control element. This control element may only serve to safely direct water or wind in order to control run-off and prevent infiltration into the building structure. The marble and granite stone according to literature ^[2] are

heat resistant. Therefore the tiles made out of powder will reflect sunlight hence increasing the surface albedo. Also Marble being white is very good reflector.



Figure 27: Cladding tiles on roof

The optimum percent replacement is used to form cladding tiles and decoration pieces using different moulds generally used in commercial markets. Tiles and objects made are shown below:



Figure 28: Different variety of cladding tiles made using 50% replacement



Figure 29: Filling the molds for making tiles



Figure 30: Tiles by adding different dyes



Figure 31: Tile for decoration



Figure 32: Ashtray (50%replacement)

4.4. COMMERCIALIZATION/ IMPLEMENTATION:

In Pakistan, the project is specific to SMEs related to cement and marble industries. Some bigger industries are Pakistan Onyx Marble and Pakistan Stone Development Company.

The implementation of this process of marble waste reuse can be done in the following way:

- Team development;
- Costing and Environmental feasibility of the technique
- General quotation development;
- Meeting consultancies and proposing them the idea; so they can help in implementing the process in various organizations facing waste management issues;

- Contacting various related SMEs and presenting them the economic, social and environmental benefits of the project
- Online Marketing of this can be done by developing websites and advertising it on various industrial forums.
- A free model of this project will be made for one small scale marble factory for display as a sample

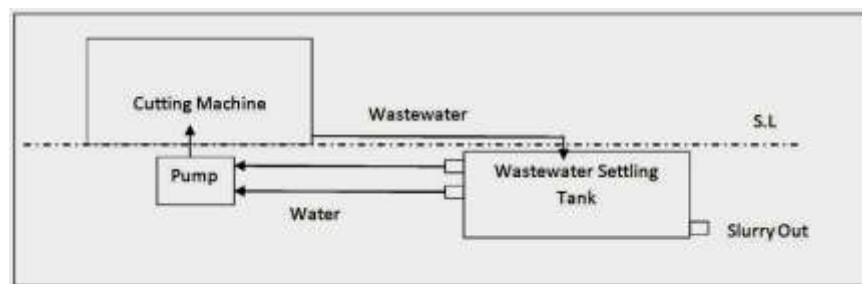


Figure 33: sample model

This sustainable project will improve environment, reduces health hazards due to marble slurry pollution and support sustainable industrial growth. Commercialization of such green projects is quite difficult. We can commercialize this project with the help of government and nongovernmental organizations (NGOs) working in Pakistan. The following organizations can help us in promoting this project

- Pakistan Green Building Council which can utilize these cladding tiles in green buildings.
- PASDEC (Pakistan Stone Development Company) has recently started a project named as marble city Risalpur, where they are using better technologies to reduce quarry losses and provide services of wastewater treatment plant and waste collection management system.
- Environmental Protection Agency.

CHAPTER 5: CONCLUSION AND RECOMMENDATIONS

5.1. CONCLUSION:

On the basis of the experimental study that has been carried out, the following conclusions can be drawn;

1. Treatment of marble water required NTU of below 50 which we achieved efficiently.
2. Settling of wastewater without alum took 240 minutes while with alum it took 90 minutes.
3. turbidity tests carried out to find the optimum dosage of sludge in wastewater Resulted:
 - ✓ For granite water treatment, ratio of sludge to sample is 1:1
 - ✓ For marble water treatment, ratio of sludge to sample is 0.5:1
4. Prisms of different compositions of marble powder, sand and cement were made and then tested for their strength.
5. Required compression strength: >1500 psi
6. Standard used: Indian standard.
7. Required strength for both marble and granite is achieved for 75% and 50% replacement of sand.

Introducing cleaner production to use sludge from Rawal lake water treatment plant is cost effective way of treating the marble factory waste and it saves time. Optimum ratio of sludge to Marble sample=0.5:1 will result in getting

below 50 NTU in 90 mins approx. This Means To Treat 1L Of Marble Wastewater We Need 0.5L Of Sludge That Has 17.5 mg Of Alum. Similarly Optimum ratio of sludge to Granite sample=0.5:1 will result in getting below 50 NTU in 120 mins approx. This Means To Treat 1L Of Granite Wastewater We Need 1L Of Sludge That Has 35 mg Of Alum. Treating marble wastewater requires half amount of sludge and almost 1.3 times lesser time to get <50 NTU as compared to granite wastewater.

Optimum percent replacement of sand with marble that gives >1500psi compression strength are 75% and 50%. Granite shows relatively more strength at the same replacement ratios due to the higher strength of natural granite stone and the apparent stronger bond with cement paste. These ratios have been used to prepare tiles and decoration pieces.

The project resulted in developing industry-university relationship.

5.2. ECONOMICS:

5.2.1. Rate of materials used:

Black Cement: Rs. 12 per Kg

White Cement: Rs. 25 per Kg

Sand: Rs. 3 for 2Kg

Color (local): Rs. 200 per Kg

Cost of 1 tile at Askari is for Rs. 12 (Black Cement, Sand and Crush)

Size: (12 x 12 x 1) inch

1 tile weighs 3000g

Cost of 1 tile with % replacement = 50%

Table 9: Marble cost calculations for 50% replacement

| Parameter | Cement | Marble | Sand |
|-----------------------------|-------------------|--------|--------------------------------|
| Ratio | 1 | 1 | 1 |
| Weigh (g) | 1000g | 1000g | 1000g |
| Cost of weighed sample (Rs) | 12 (black cement) | - | 1 (actually it costs Rs. 0.67) |
| Cost of weighed sample (Rs) | 25 (white cement) | - | 1 (actually it costs Rs. 0.67) |

Black Cement: Cost of 1 tile (12 x 12 x 1) inch, made from black cement, marble and sand (according to 2013 data) = Rs. 13 (actually Rs. 12.67)

White Cement: Cost of 1 tile (12 x 12 x 1) inch, made from white cement, marble and sand (according to 2013 data) = Rs. 26 (actually Rs. 25.67)

Cost of 1 tile with % replacement = 75%

Table 10: Marble cost calculations for 75% replacement

| | Cement | Marble | Sand |
|-----------------------------|-------------------|--------|--------------------------------|
| Ratio | 1 | 1.5 | 0.5 |
| Weigh (g) | 1000g | 1500g | 500g |
| Cost of weighed sample (Rs) | 12 (black cement) | - | 1 (actually it costs Rs. 0.33) |
| Cost of weighed sample (Rs) | 25 (white cement) | - | 1 (actually it costs Rs. 0.33) |

Black Cement: Cost of 1 tile (12 x 12 x 1) inch, made from black cement, marble and sand (according to 2013 data)= Rs. 13 (actually Rs. 12.33)

White Cement: Cost of 1 tile (12 x 12 x 1) inch, made from white cement, marble and sand (according to 2013 data) = Rs. 26 (actually Rs. 25.33)

5.2.2. Granite

Cost of 1 tile with % replacement = 50%

Table 11: Granite cost calculations for 50% replacement

| | Cement | Granite | Sand |
|------------------------------------|-------------------|---------|--------------------------------|
| Ratio | 1 | 1 | 1 |
| Weigh (g) | 1000g | 1000g | 1000g |
| Cost of weighed sample (Rs) | 12 (black cement) | - | 1 (actually it costs Rs. 0.67) |
| Cost of weighed sample (Rs) | 25 (white cement) | - | 1 (actually it costs Rs. 0.67) |

Black Cement: Cost of 1 tile (12 x 12 x 1) inch, made from black cement, Granite and sand (according to 2013 data) = Rs. 13 (actually Rs. 12.67)

White Cement: Cost of 1 tile (12 x 12 x 1) inch, made from white cement, Granite and sand (according to 2013 data) = Rs. 26 (actually Rs. 25.67)

Cost of 1 tile with % replacement = 75%

Table 12: Granite cost calculations for 75% replacement

| | Cement | Granite | Sand |
|------------------------------------|-------------------|---------|--------------------------------|
| Ratio | 1 | 1.5 | 0.5 |
| Weigh (g) | 1000g | 1500g | 500g |
| Cost of weighed sample (Rs) | 12 (black cement) | - | 1 (actually it costs Rs. 0.33) |
| Cost of weighed sample (Rs) | 25 (white cement) | - | 1 (actually it costs Rs. 0.33) |

Black Cement: Cost of 1 tile (12 x 12 x 1) inch, made from black cement, Granite and sand (according to 2013 data) = Rs. 13 (actually Rs. 12.33)

White Cement: Cost of 1 tile (12 x 12 x 1) inch, made from white cement, Granite and sand (according to 2013 data) = Rs. 26 (actually Rs. 25.33)

5.3. PROBLEMS FACED:

1. Collection of sludge sample during working hours.
2. Transportation and storage of wet marble waste.

3. Unavailability of apparatus in IESE labs.
4. Dumping of prism/tiles/blocks broken after different strength tests.
5. Limited apparatus in NICE structure lab. Only one UTM machine for whole NUST.

5.4. OTHER PROPOSITIONS:

1. Making of cladding tiles, tough tiles for flooring purposes.
2. Producing decoration pieces.
3. Sculpture making.
4. Use in ceramic industry.
5. Enhancement of reflective property of common tiles.

5.5. FUTURE PROSPECTS:

1. Use of the marble waste in the hydraulic concrete.
2. Incorporation of marble sludge in industrial building.
3. Eco-blocks or cement bricks formulation.
4. Bringing cost efficiency to the ceramic market.
5. Economical value addition to solid waste.

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APPENDIX 1
UNITS OF MEASUREMENT

UNITS OF MEASUREMENT:

| 1000^m | 10^n | Prefix | Symbol | Short scale | Long scale | Decimal |
|---------------|------------|--------|--------|---------------|---------------|-----------------------------------|
| 1000^8 | 10^{24} | yotta- | Y | Septillion | Quadrillion | 1 000 000 000 000 000 000 000 000 |
| 1000^7 | 10^{21} | zetta- | Z | Sextillion | Trilliard | 1 000 000 000 000 000 000 000 |
| 1000^6 | 10^{18} | exa- | E | Quintillion | Trillion | 1 000 000 000 000 000 000 |
| 1000^5 | 10^{15} | peta- | P | Quadrillion | Billiard | 1 000 000 000 000 000 |
| 1000^4 | 10^{12} | tera- | T | Trillion | Billion | 1 000 000 000 000 |
| 1000^3 | 10^9 | giga- | G | Billion | Milliard | 1 000 000 000 |
| 1000^2 | 10^6 | mega- | M | Million | | 1 000 000 |
| 1000^1 | 10^3 | kilo- | k | Thousand | | 1 000 |
| $1000^{2/3}$ | 10^2 | hecto- | h | Hundred | | 100 |
| $1000^{1/3}$ | 10^1 | deca- | da | Ten | | 10 |
| 1000^0 | 10^0 | (none) | (none) | One | | 1 |
| $1000^{-1/3}$ | 10^{-1} | deci- | d | Tenth | | 0.1 |
| $1000^{-2/3}$ | 10^{-2} | centi- | c | Hundredth | | 0.01 |
| 1000^{-1} | 10^{-3} | milli- | m | Thousandth | | 0.001 |
| 1000^{-2} | 10^{-6} | micro- | μ | Millionth | | 0.000 001 |
| 1000^{-3} | 10^{-9} | nano- | n | Billionth | Milliardth | 0.000 000 001 |
| 1000^{-4} | 10^{-12} | pico- | p | Trillionth | Billionth | 0.000 000 000 001 |
| 1000^{-5} | 10^{-15} | femto- | f | Quadrillionth | Billiardth | 0.000 000 000 000 001 |
| 1000^{-6} | 10^{-18} | atto- | a | Quintillionth | Trillionth | 0.000 000 000 000 000 001 |
| 1000^{-7} | 10^{-21} | zepto- | z | Sextillionth | Trilliardth | 0.000 000 000 000 000 000 001 |
| 1000^{-8} | 10^{-24} | yocto- | y | Septillionth | Quadrillionth | 0.000 000 000 000 000 000 000 001 |

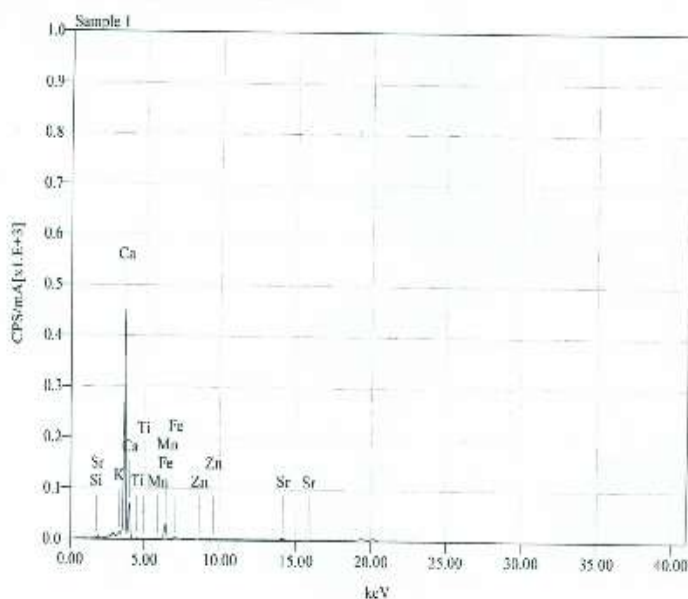
Weight and Volume Conversion Chart

| Weight | | Volume | |
|----------------|----------------|----------------|----------------|
| <i>Lb = Kg</i> | <i>Kg = Lb</i> | <i>Gal = L</i> | <i>L = Gal</i> |
| 1 = 0.45 | 1 = 2.21 | 1 = 3.79 | 1 = 0.26 |
| 2 = 0.91 | 2 = 4.41 | 2 = 7.57 | 2 = 0.53 |
| 3 = 1.36 | 3 = 6.61 | 3 = 11.35 | 3 = 0.79 |
| 4 = 1.81 | 4 = 8.82 | 4 = 15.14 | 4 = 1.06 |
| 5 = 2.27 | 5 = 11.02 | 5 = 18.93 | 5 = 1.32 |
| 6 = 2.72 | 6 = 13.23 | 6 = 22.71 | 6 = 1.58 |
| 7 = 3.18 | 7 = 15.43 | 7 = 26.5 | 7 = 1.85 |
| 8 = 3.63 | 8 = 17.46 | 8 = 30.28 | 8 = 2.11 |
| 9 = 4.08 | 9 = 19.84 | 9 = 34.16 | 9 = 2.38 |
| 10 = 4.54 | 10 = 22.05 | 10 = 37.94 | 10 = 2.64 |
| 50 = 22.68 | 50 = 110.23 | 50 = 189.70 | 50 = 13.2 |
| 100 = 45.36 | 100 = 220.46 | 100 = 379.40 | 100 = 26.4 |

| Kgf/cm ² | Mpa | bar | kpa | mbar | psi | MmAq MmH ₂ O | Torr mmHg | atm |
|---------------------|-----------|-----------|----------|-----------|-----------|----------------------------|--------------|-----------|
| 1 | 0.0980665 | 0.980665 | 98.0665 | 980.665 | 14.2231 | 10,000.00 | 735.294 | 0.98749 |
| 10.1972 | 1 | 10 | 1,000.00 | 10,000.00 | 145.036 | 101971.6 | 7500.60 | 9.8692 |
| 1.01972 | 0.1 | 1 | 100 | 1,000.00 | 14.5036 | 10197.16 | 750.062 | 0.98692 |
| 0.010197 | 0.001 | 0.01 | 1 | 10 | 0.145 | 101.9716 | 7.50062 | 0.009869 |
| 0.0010197 | 0.0001 | 0.001 | 0.1 | 1 | 0.0145 | 10.19716 | 0.750062 | 0.0009869 |
| 0.07031 | 0.006895 | 0.06895 | 6.895 | 68.95 | 1 | 703.08 | 51.7157 | 0.06805 |
| 0.0001 | 0.0000098 | 0.000098 | 0.009806 | 0.098 | 0.0014223 | 1 | 0.073529 | 0.0000967 |
| 0.0013595 | 0.0001332 | 0.0013332 | 0.13332 | 1.33321 | 0.0193364 | 13.5951 | 1 | 0.0013157 |
| 1.033228 | 0.101325 | 1.0232506 | 101.325 | 1013.2506 | 14.69574 | 10,332.28 | 760.00 | 1 |

APPENDIX 2

XRF RESULTS



File Name : D:\Documents and Settings\Admin Khan\Desktop\EDS Samples\Sample 1.ed2
 Name : Sample 1
 Sample Name: Sample 1
 Date : 10/25/2012 10:39:39 AM

Acquisition Parameter
 Tube Voltage: 20.000 kV Tube Current: 1.000 mA
 Real Time : 40.32 min. Dead Time : 27 %
 Live Time : 30.00 sec. Counting Rate: 16328 Counts/sec.
 Preset : Live Time 30.00 sec.
 Energy Range: 0 - 41 keV PHA Mode: T2
 Pass : AIR

Optical Parameter
 Collimator : 7.000 mm Filter :

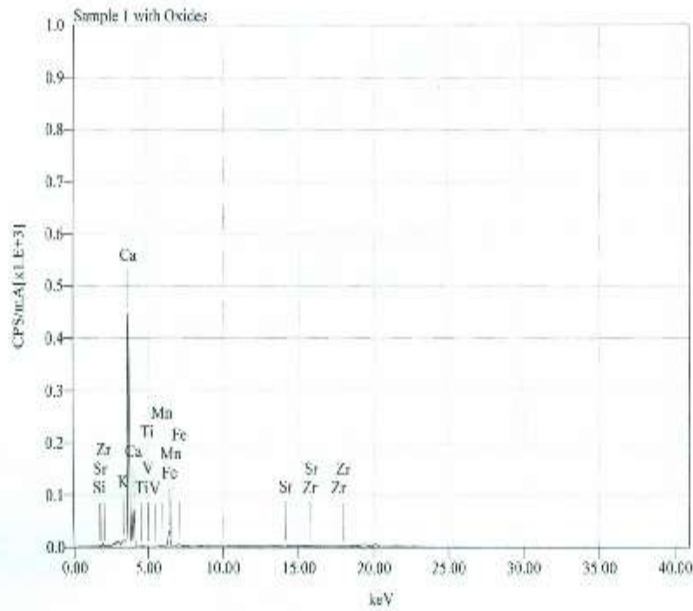
Qualitative result
 Analysis Elements: Si, K, Ca, Ti, Mn, Fe, Zn, Sr
 Fitting Coefficient: 0.0697

Quantitative condition

Quantitative result

| Element | wt% | wol% | Sigma | Intensity | E ratio | Line | Type |
|---------|---------|---------|---------|-----------|-----------|------|------|
| 14 Si | 18.3364 | 24.4228 | 10.0811 | 316 | 0.0942353 | K | |
| 19 K | 1.0658 | 1.0221 | 0.0029 | 2108 | 0.0029068 | K | |
| 20 Ca | 77.1636 | 72.1312 | 0.0374 | 211995 | 0.2229221 | K | |
| 22 Ti | 0.3578 | 0.2800 | 0.0353 | 158 | 0.0004772 | K | |
| 25 Mn | 0.0864 | 0.0530 | 0.0179 | 396 | 0.0001680 | K | |
| 26 Fe | 2.8017 | 1.8811 | 0.0157 | 16039 | 0.0059251 | K | |
| 30 Zn | 0.0356 | 0.0204 | 0.0112 | 333 | 0.0001150 | K | |
| 38 Sr | 0.1529 | 0.0654 | 0.0125 | 2079 | 0.0010998 | K | |

| Name | From | To | Center | Gross(CPS) | Net(CPS) | Gross(Counts) | Net(Counts) |
|------|------|----|--------|------------|----------|---------------|-------------|
|------|------|----|--------|------------|----------|---------------|-------------|



File Name : C:\Documents and Settings\Anjed Khan\Desktop\UG Samples\Sample 1 with Oxides.sp2
 Memo : Sample 1 with Oxides
 Sample Name: Sample 1 with Oxides
 Date : 10/25/2012 10:37:19 AM

Acquisition Parameter
 Tube Voltage: 30.000 kV Tube Current : 1.000 mA
 Real Time : 40.77 sec. Dead Time : 26 %
 Live Time : 30.00 sec. Counting Rate: 16374 Counts/sec.
 Preset : Live Time 30.00 sec.
 Energy Range: 0 - 41 keV PEA Mode : T2
 Pass : AIR

Optical Parameter
 Collimator : 7.000 mm Filter :

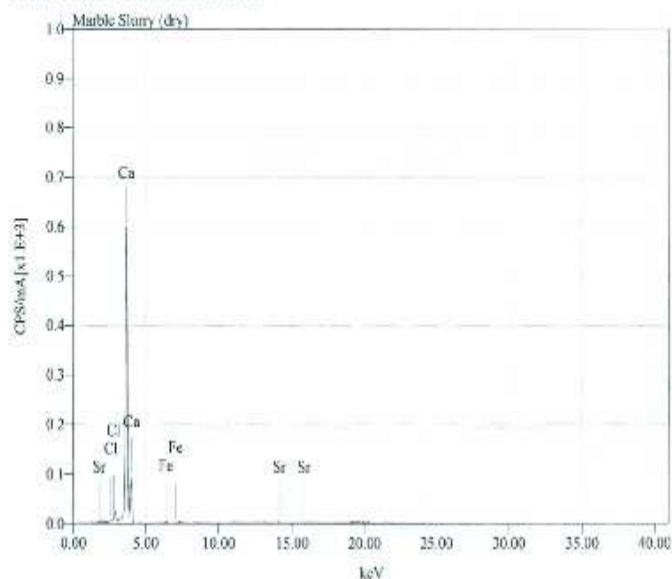
Qualitative result
 Analysis Elements: Si, K, Ca, Ti, V, Mn, Fe, Sr, Zr
 Fitting Coefficient: 0.0713

Quantitative condition

Quantitative result:

| Element | wt% | mol% | Sigma | Intensity | K ratio | Line | Type |
|----------|---------|---------|---------|-----------|-----------|------|------|
| 14 SiO2 | 27.4351 | 26.6365 | 19.2314 | 275 | 0.0296884 | K | |
| 19 K2O | 0.8539 | 0.5288 | 0.0482 | 2032 | 0.0035763 | K | |
| 20 CaO | 68.8690 | 71.6386 | 0.0366 | 212057 | 0.2229672 | K | |
| 22 TiO2 | 0.2947 | 0.2152 | 0.0367 | 664 | 0.0004183 | K | |
| 23 V2O5 | 0.0578 | 0.0165 | 0.0317 | 151 | 0.0000831 | K | |
| 25 MnO | 0.0837 | 0.0573 | 0.0142 | 442 | 0.0001876 | K | |
| 26 Fe2O3 | 2.3096 | 0.4437 | 0.0138 | 16548 | 0.0051132 | K | |
| 38 SrO | 0.0367 | 0.0544 | 0.0009 | 2020 | 0.0010689 | K | |
| 40 ZrO2 | 0.0146 | 0.0069 | 0.0121 | 281 | 0.0001680 | K | |

| Name | From | To | Center | Gross (CPS) | Net (CPS) | Gross (Counts) | Net (Counts) |
|------|------|----|--------|-------------|-----------|----------------|--------------|
|------|------|----|--------|-------------|-----------|----------------|--------------|



File Name : C:\Documents and Settings\amjad Khan\Desktop\UG Samples\Marble Slurry (dry).ap2
 Name : Marble Slurry (dry)
 Sample Name: Marble Slurry (dry)
 Date : 11/15/2012 10:23:42 AM

Acquisition Parameter
 Tube Voltage: 30.000 kV Tube Current : 1.000 mA
 Real Time : 42.36 sec. Dead Time : 29 %
 Live Time : 30.00 sec. Counting Rate: 18303 Counts/sec.
 Preset : Live Time 30.00 sec.
 Energy Range: 0 - 41 keV FHR Mode : F2
 Pass : AIR

Optical Parameter
 Collimator : 7.000 mm Filter :

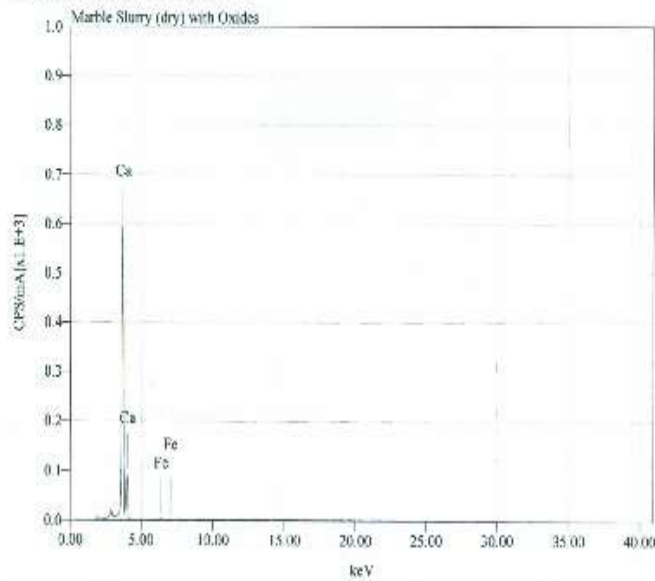
Qualitative result
 Analysis Elements: Cl, Ca, Fe, Sr
 Fitting Coefficient: 0.0932

Quantitative condition

Quantitative result

| Element | wt% | mol% | Sigma | Intensity | K ratio | Line | Type |
|---------|---------|---------|--------|-----------|-----------|------|------|
| 17 Cl* | 0.8291 | 0.8379 | 0.1674 | 720 | 0.0027113 | K | |
| 20 Ca | 98.6439 | 98.6984 | 0.0863 | 283455 | 0.2980655 | K | |
| 26 Fe | 0.4707 | 0.3280 | 0.0480 | 2127 | 0.0007859 | K | |
| 38 Sr | 0.0563 | 0.0258 | 0.0368 | 626 | 0.0003313 | K | |

| Name | From | To | Center | Gross(CPS) | Net(CPS) | Gross(Counts) | Net(Counts) |
|------|------|----|--------|------------|----------|---------------|-------------|
|------|------|----|--------|------------|----------|---------------|-------------|



File Name : C:\Documents and Settings\Anjad Khan\Desktop\XRF Samples\Marble Slurry (dry) with Oxides.
 Name : Marble Slurry (dry) with Oxides
 Sample Name: Marble Slurry (dry) with Oxides
 Date : 11/19/2012 10:26:47 AM

Acquisition Parameter
 Tube Voltage: 30.000 kV Tube Current: 1.000 mA
 Beam Time : 42.35 sec. Dead Time : 21 %
 Live Time : 33.00 sec. Counting Rate: 18319 Counts/sec.
 Preset : Live Time 30.00 sec.
 Energy Range: 0 - 41 keV EDS Mode : ED
 Pass : AIR

Optical Parameter
 Collimator : 7.000 mm Filter :

Qualitative result
 Analysis Elements: Ca, Fe
 Fitting Coefficient: 0.0768

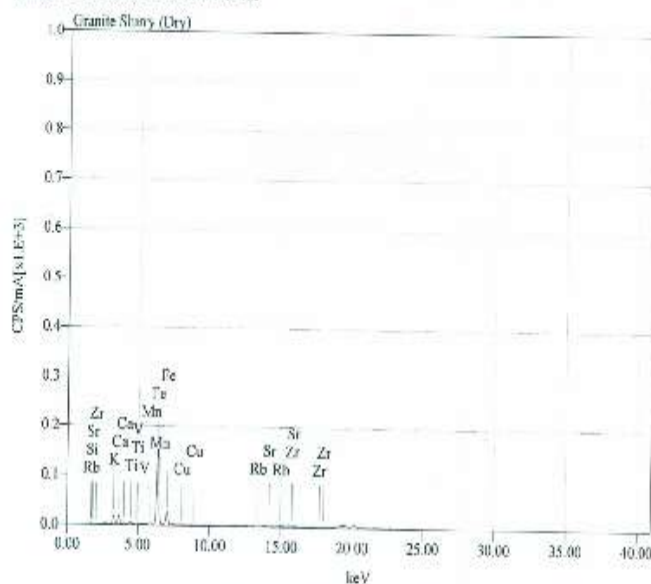
Quantitative condition

| Element | wt% | wol% | Stoich | Intensity | Z ratio | Line | Type |
|----------|---------|---------|--------|-----------|-----------|------|------|
| 20 CaO | 99.5854 | 99.8540 | 0.0768 | 293663 | 0.2982844 | K | |
| 26 Fe2O3 | 0.4146 | 0.1450 | 0.0595 | 2009 | 0.0007421 | K | |

| Name | From | To | Center | Gross(CPS) | Net(CPS) | Gross(Counts) | Net(Counts) |
|------|------|----|--------|------------|----------|---------------|-------------|
|------|------|----|--------|------------|----------|---------------|-------------|

Print: 11/29/2012 11:20:43 AM

1/1



File Name : C:\Documents and Settings\Amjad Khan\Desktop\00 Samples\Granite Slurry (Dry).sp2
 Memo : Granite Slurry (Dry)
 Sample Name: Granite Slurry (Dry)
 Date : 11/15/2012 10:31:47 AM

Acquisition Parameter
 Tube Voltage: 30.000 kV Tube Current : 1.000 mA
 Real Time : 36.96 sec. Dead Time : 13 %
 Live Time : 30.00 sec. Counting Rate: 10943 Counts/sec.
 Product : Live Time 30.00 sec.
 Energy Range: 0 - 41 keV PMA Mode : TE
 Base : Ahk

Optical Parameter
 Collimator : 7.500 mm Filter :

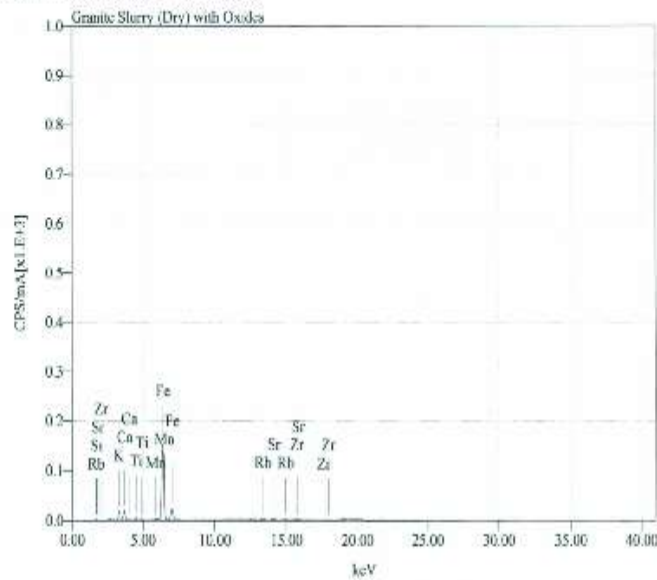
Qualitative result
 Analyzed Elements: Si, S, Ca, Ti, V, Mn, Fe, Cu, Rb, Sr, Zr
 Fitting Coefficient: 0.424

Quantitative condition

Quantitative result

| Element | wt% | mol% | Sigma | Intensity | K ratio | Line | Type |
|---------|---------|---------|---------|-----------|-----------|------|------|
| 14 Si | 61.5507 | 72.3695 | 30.1530 | 629 | 0.0679493 | K | |
| 19 K | 17.1415 | 10.2537 | 0.1207 | 8148 | 0.0123407 | K | |
| 20 Ca | 7.2442 | 6.5453 | 0.0723 | 8763 | 0.0082150 | K | |
| 22 Ti | 1.1985 | 0.0269 | 0.0260 | 2694 | 0.0016960 | K | |
| 23 V | 0.0797 | 0.0516 | 0.0210 | 223 | 0.0001723 | K | |
| 25 Mn | 0.2642 | 0.1749 | 0.0133 | 1305 | 0.0005541 | K | |
| 26 Fe | 16.0585 | 9.4954 | 0.0130 | 86129 | 0.0318172 | K | |
| 29 Cu | 0.0550 | 0.0389 | 0.0129 | 328 | 0.0001141 | K | |
| 31 Rb | 0.1822 | 0.0704 | 0.0144 | 1885 | 0.0008209 | K | |
| 38 Sr | 0.2398 | 0.0900 | 0.0130 | 2260 | 0.0011906 | K | |
| 40 Zr | 0.2500 | 0.0923 | 0.0160 | 2510 | 0.0014992 | K | |

| Name | From | To | Center | Gross (CPS) | Net (CPS) | Gross (Counts) | Net (Counts) |
|------|------|----|--------|-------------|-----------|----------------|--------------|
|------|------|----|--------|-------------|-----------|----------------|--------------|



File Name : C:\Documents and Settings\Anjad Khan\Desktop\AUG Samples\Granite Slurry (Dry) with Oxides.
 Memo : Granite Slurry (Dry) with Oxides
 Sample Name: Granite Slurry (Dry) with Oxides
 Date : 11/15/2012 10:30:01 AM

Acquisition Parameter
 Tube Voltage: 30.000 KV Tube Current : 1.000 nA
 Read Time : 35.94 sec. Read Time : 16.3
 Live Time : 30.00 sec. Counting Rate: 10962 Counts/sec.
 Preset : Live Time 30.00 sec.
 Energy Range: 0 - 41 keV PMA Mode : T2
 Peak : NIB

Optical Parameter
 Collimator : 7.000 mm Filter :

Qualitative result
 Analysis Elements: Si, K, Ca, Ti, Mn, Fe, Rb, Sr, Zr
 Fitting Coefficient: 0.1531

Quantitative condition

Quantitative result

| Element | wt% | mol% | Sigma | Intensity | K ratio | Line | Type |
|----------|---------|---------|---------|-----------|-----------|------|------|
| 14 SiO2 | 63.0804 | 68.5490 | 15.9328 | 778 | 0.084165 | K | |
| 19 K2O | 5.5333 | 3.7615 | 0.1638 | 8146 | 0.0143373 | K | |
| 20 CaO | 3.7542 | 4.2871 | 0.1401 | 8661 | 0.0091070 | K | |
| 22 TiO2 | 3.6610 | 0.5298 | 0.1159 | 2796 | 0.0017601 | K | |
| 25 MnO | 3.1116 | 0.1308 | 0.0064 | 1282 | 0.0054886 | K | |
| 26 Fe2O3 | 6.6624 | 2.6718 | 0.0062 | 66217 | 0.0318501 | L | |
| 37 Rb2O | 3.0511 | 0.3175 | 0.0045 | 1979 | 0.0008618 | K | |
| 38 SrO | 0.9684 | 0.0623 | 0.0048 | 2251 | 0.0011907 | K | |
| 40 ZrO2 | 0.0775 | 0.0403 | 0.1064 | 2337 | 0.0013958 | K | |

| Name | From | To | Center | Gross(CPS) | Net(CPS) | Gross(Counts) | Net(Counts) |
|------|------|----|--------|------------|----------|---------------|-------------|
|------|------|----|--------|------------|----------|---------------|-------------|

APPENDIX 3
VISIT TO KOHSAR INDUSTRY AND SAMPLE
COLLECTION

VISIT TO KOHSAR INDUSTRY AND SAMPLE COLLECTION





APPENDIX 4
ASTM STANDARD C648



Standard Test Method for Breaking Strength of Ceramic Tile¹

This standard is issued under the fixed designation C 648; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ϵ) indicates an editorial change since the last revision or reapproval.

1. Scope

1.1 This test method covers the determination of the breaking strength of glazed ceramic wall tile, ceramic mosaic tile, quarry tile, and paver tile, having a facial area of at least 1 in.² (6.4 cm²).

1.2 The values stated in inch-pound units are to be regarded as the standard. The metric equivalents of inch-pound units may be approximate.

1.3 *This standard does not purport to address all of the safety concerns, if any, associated with its use. It is the responsibility of the user of this standard to establish appropriate safety and health practices and determine the applicability of regulatory limitations prior to use.*

2. Referenced Documents

2.1 ASTM Standards:

C 242 Terminology of Ceramic Whitewares and Related Products²

E 178 Practice for Dealing with Outlying Observations³

3. Terminology

3.1 Definitions:

3.1.1 For definitions of the types of tile listed in Section 1, refer to Terminology C 242.

4. Summary of Test Method

4.1 The test method consists of supporting the tile on the ends of three cylindrical rods, or on three ball bearings for tiles having an actual facial area of 9 in.² (58 cm²) (or less), arranged in an equilateral triangle, and applying force at a definite rate to the center of the tile, which coincides with the center of the triangular support, until the specimen breaks.

NOTE 1—The size (facial dimensions) of otherwise equal tile affects the value of breaking strength obtained by this test method. For instance, a 6 by 6-in. (152 by 152-mm) tile equal in mature body properties and thickness to a 4½ by 4½-in. (108 by 108-mm) tile will have a breaking strength higher than the 4½ by 4½-in. tile.

¹ This test method is under the jurisdiction of ASTM Committee C-21 on Ceramic Whitewares and Related Products and is the direct responsibility of Subcommittee C21.06 on Ceramic Tile.

Current edition approved July 10, 1998. Published January 1999. Originally published as C 648 – 70. Last previous edition C 648 – 84 (1994).

² Annual Book of ASTM Standards, Vol 15.02.

³ Annual Book of ASTM Standards, Vol 14.02.

5. Significance and Use

5.1 The test method provides means for establishing whether or not a lot of ceramic tile meets the strength requirements which may appear in tile specifications. Tile strength is the force in pounds-force (or newtons), as read from the pressure gage, necessary to cause the tile to break.

6. Apparatus

6.1 *Specimen-Support*, consisting of a 5½ by 5½ by 1-in. (139.7 by 139.7 by 25.4-mm) steel block. Three ¼-in. (6.4-mm) diameter and ¼-in. (9.5-mm) deep tapped holes are arranged in an equilateral triangle having 3-in. (76.2-mm) sides and whose circumscribed circle has a radius of 1.732 in. (44.0 mm). The equilateral triangle is located on the steel block so that its center coincides with that of the steel block and one side of the triangle runs parallel to one side of the steel block. Three ¼-in. diameter steel rods, 1½ in. with (34.9 mm) long, one end threaded for a distance of ¼ in. with the same ¼-in. thread as the tapped holes, and the other end ground flat, fit into the three holes. In a similar manner, a second set of three ¼-in. diameter steel rods, but ¼ in. (19.0 mm) long only, are threaded on one end for a distance of ¾-in., are ground flat on the other end and fit three other ¼-in. tapped holes. The three ¼-in. holes, tapped for a distance of ¾ in. to receive this second set of steel rods are located at the apexes of an equilateral triangle having 1½-in. (38.1-mm) long sides and whose circumscribed circle has a radius of 0.866 in. (22.0 mm). This second equilateral triangle is located within the first one in such a manner that its circumscribed circle is identical with the inscribed circle of the first triangle, and its sides are parallel to those of the larger triangle. The ball bearing support consists of three ¼-in. (3.2-mm) ball bearings arranged in an equilateral triangle, with sides of 14 mm concentric and parallel with the other triangles. The bearings are embedded in the block to the extent of half their volume. The steel block has four more ¼-in. tapped holes to receive socket head cap screws for holding four registry stops. One of these holes is located in the center of one side, ¼ in. in from the edge. Another of these is located in the center of an adjacent side ¼ in. from the edge. The other two are located on an adjacent side, ¼ in. in from the edge, spaced 2 in. (50.8 mm) apart, with one located 1½ in. (44.4 mm) from one of the corners of the first side. The two single hole registry

stops are used with the ball bearing support. The double hole registry stop and the adjacent edge single stop are to be used with the sets of rod supports. Three of the registry stops designed for use with the cylindrical rod supports consist of $\frac{1}{8}$ -in. (3.2-mm) thick steel strips, $\frac{3}{4}$ in. wide, 2 in. long, with a $\frac{1}{4}$ -in. wide slot extending for the full 2-in. length of the strip. The slot is open on one end and the other end of each steel strip has a $\frac{3}{4}$ in. wide and $1\frac{1}{2}$ in. high projection, $\frac{1}{8}$ in. thick, which will contact the edges of the tile when in proper position for use. The other two registry stops, designed for use with the ball bearing support, have exactly the same dimensions except for their projection height which is $\frac{3}{8}$ in. The projection is obtained by making the steel strips actually $3\frac{1}{2}$ in. (88.9 mm) long and bending them at right angles to obtain the $1\frac{1}{2}$ -in. or $\frac{3}{4}$ -in. (38.1-mm or 15.9-mm) high projection. Each of the three registry stops can be fastened with washer and socket head cap screw through its slot in any one of numerous positions. Being adjustable, they serve as registry stops for the different sizes of

tile which may be broken over these supports. For tile of dimensions $4\frac{1}{4}$ in. (107.9 mm) or smaller, the projections will be located on that end of the strip nearest to the support block; for tile having dimensions larger than $4\frac{1}{4}$ in., the strips are reversed so that the projections are located on the end of the steel strips away from the support block (see Fig. 1). For certain mosaic tiles with dimensions of less than 1 in. (25.4 mm) length, it is necessary to reverse the position of the registry stops so that the tile is supported with the slot ends in order to avoid interference with the force applicator.

6.2 *Force Applicator*—Force is applied in the exact center of the triangular support by means of a $\frac{1}{2}$ -in. (12.7-mm) diameter stainless steel ball bearing, countersunk to a depth of $\frac{1}{16}$ in. (6.7 mm) into one end of a 1-in. (25.4-mm) diameter and 2-in. long steel rod. A $\frac{3}{32}$ -in. (2.4-mm) thick retaining collar prevents the ball from falling out of its recess and is fastened to the steel rod by four $\frac{3}{32}$ -in. 3-48 NC screws, $\frac{3}{8}$ in. (9.5 mm) long, with countersunk heads. The opposite end of

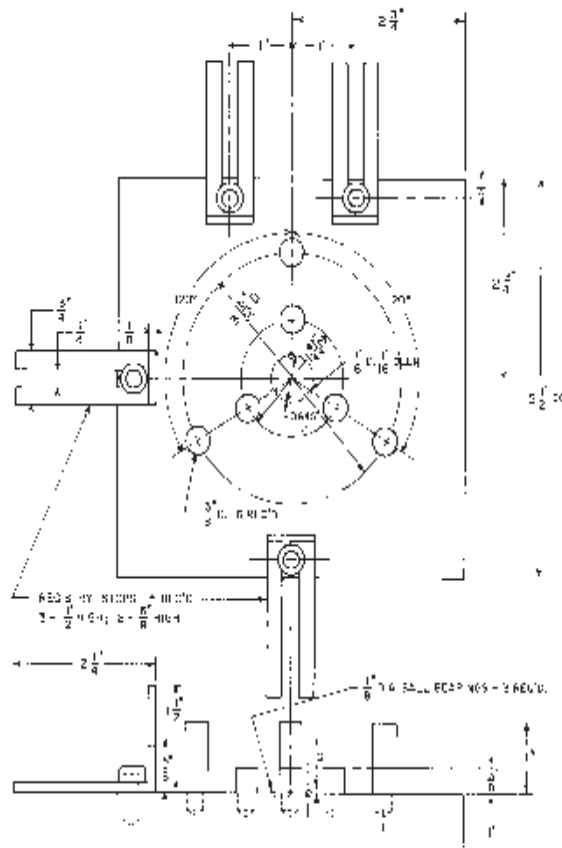


FIG. 1 Support Block Assembly (See Table 1)

position of the tile. Except for minor size variations, the registry stops will now locate every tile of the sample in the relatively same spot on the support, so that force is applied in the approximate center of each tile.

8.4 Position a safety shield around the apparatus so that no person or property will be injured from possible contact with broken tile. The use of a safety shield is mandatory in testing the breaking strength of ceramic mosaic tile, which can break into shrapnel-like fragments.

8.5 Whenever the approximate force required to break the particular type of tile is not known, it is necessary to break several tile prior to starting the test in order to establish the range of the gage to be used. Install the proper gage and set the peak-load indicator to zero.

8.6 Apply force at the rate of 800 to 1100 lbf (3600 to 4900 N)/min until the tile actually breaks into two or more pieces. Read the force on the gage by interpolation to the nearest 1 lbf (4 N). Record this pressure and reset the gage to zero.

8.7 Remove any debris from the surface of the steel block with a brush. Place the next tile on the triangular support with two adjacent sides in contact with the registry stops and repeat the operation until all of the tile in the sample have been broken.

9. Calculation

9.1 Average the breaking forces for all of the tile in the sample.

9.2 If any one individual breaking force appears to deviate markedly from the others of the sample, it may be eliminated

before the average is calculated only after the criteria in Practice E 178 have been applied and justify elimination.

9.3 Should it become necessary to eliminate more than 20 % of the individual values in a sample in accordance with 9.2, repeat the test on sample containing twice the number of specimens in the original one. In that case, only breaking forces from the repeat test shall be used to arrive at the average strength of the sample.

10. Report

10.1 Report the following information:

10.1.1 Name and model number of the testing machine,

10.1.2 Range of the gage used,

10.1.3 Identification of the triangular support used in testing, by stating the length of its sides,

10.1.4 Type of tile tested, whether glazed or unglazed.

10.1.5 Number of specimens in the sample; also state whether it is a repeat test, and

10.1.6 Average strength in pounds-force (or newtons) of the sample.

11. Precision and Bias

11.1 The maximum difference between breaking strengths determined by laboratories testing tile from the same lots was 20 % of the lower value, and the difference was less than 5 % for 50 % of the lots tested. An overall difference of 7½ % existed between laboratories in a test series of 19 lots of wall tile.

12. Keywords

12.1 breaking strength; ceramic tile

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