ADDIS ABABA UNIVERSITY

SCHOOL OF GRADUATE STUDIES

ADDIS ABABA INSITUTE OF TECHNOLOGY

DEPARTMENT OF CHEMICAL AND BIO ENGINEERING

RECYCLING PLASTER OF PARIS FROM DISCARDED GYPSUM MOULDS FOR TABOR CERAMICS PRODUCTS MANUFACTURING SHARE COMPANY

By: Tesfaye Ayele Bekele

FEBRUARY 2015

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A Thesis Submitted to the Research and Graduate School of Addis Ababa University, Addis Ababa Institute of Technology, Department of Chemical and Bio Engineering in Partial Fulfilment of the Requirements for the Attainment of the Degree of Masters of Science in Chemical and Bio Engineering Under Process Engineering Stream.

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Advisor: Ing. Gizachew Shiferaw

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ACRONYMS

3-D	Three dimension
α-hemihydrate	Alpha hemihydrate
β-hemihydrate	Beta hemihydrates
ANOVA	Analysis of variance
ASTM	American standard for testing materials
C&D	Construction and demolition
CCD	Central composite design
DSC	Differential scanning calorimetry
DTA	Differential thermal analyses
EMPDE	Educational Materials production and Distribution Enterprise
FGD	Flue gas desulfurization
kWh	Kilo watt hour
MPa	Mega pascal
POP	Plaster of Paris
rpm	Revolution per minutes
TDS	Total dissolved solid

ABSTRACT

A considerable amount of waste plaster of Paris or gypsum mould is produced in the pottery, and sanitary ware industries. Conventionally, this waste product has usually been disposed of in landfill sites and the like. This is becoming uneconomical due to the increasing cost of gypsum and particularly with diminishing productivity of some sources of gypsum, and also the introduction of landfill taxes.

This thesis research seeks to address this problem by recycling plaster of Paris from discarded gypsum mould. The study has been conducted in such a way that, first discarded gypsum mould samples collected from Tabor ceramics products factory was washed and sieved to remove dust and soluble deflocculates. The result showed 8.4% impurity content. The mole of water of crystallization of the sample before calcination was found to be 1.943 and this proved that it was in a dihydrate state since mole of water of crystallization of dihydrate gypsum is 2. Following the same procedure, calcination of the sample at a temperature of 148.5^{o}C for 2.2 hours resulted in mole of water of crystallization of 0.45 and this proves conversion to hemihydrate state since mole of water of crystallization of hemihydrate plaster is 0.5. The recycled powder regained whitish color with a rise in temperature starting from a calcination temperature of 140^{o}c.

The experimental design and statistical analysis was done by Design-Expert 7.0.0 program. From the result of analysis, maximum setting time was found to be 19.3 minutes at calcination temperature of 145^{0}c and calcination time of 3 hours. All values of water absorption are well within the working ranges of the factory. The highest value of compressive strength obtained is 5.7010 MPa at calcination temperature of 148.5^{0}C and calcination time of 2.15 hours.The optimization analysis gives a setting time value of 15 minutes, water absorption 0.25cm/minute and compressive strength value of 5.7011MPa at 148.5^{o}C calcination temperature and 2.22 hours calcination time. The result of the blending experiment shows that blending generally increases the compressive strength and a maximum of 8.6956MPa is obtained when 60% recycled plaster of paris is mixed with 40% vergin plaster of paris.

Finally, the plant is financially evaluated and its initial investment cost will be recovered fully within two operation years which makes the plant viable

1. INTRODUCTION

1.1 Background

Plaster of Paris is produced by calcining gypsum. The main deposits are found in the United States, France, West Germany, Poland, and Australia. [6] There are extensive and thick gypsum and anhydrite deposits in central Ethiopia (in the Blue Nile area), and some deposits of lesser thickness in the Mekele area of northern Ethiopia and in the southeast of the country. [23]

A large quantity of the world production of plaster of Paris is used in the ceramic industry for the manufacturing of moulds and cementitious materials for construction purpose. It is also used for the manufacturing of wall boards, dental and orthopedic work. [2]

In Tabor ceramic products manufacturing company, plaster of Paris is used for making table ware and sanitary ware moulds for casting the ceramic wares. In 2012, the total production of moulds from plaster of Paris was around 40 tons per month. After using moulds for a certain period of time, it is discarded as waste material. Plaster of Paris is imported from abroad, mainly from China and India, at high cost and therefore it is essential to study the possibility of recycling plaster of Paris. In order to develop a method of recycling, it is essential to study the physical and chemical properties of used moulds and plaster of Paris.

Besides to its economical aspect, when old dry gypsum is placed in landfills several things may occur. When the gypsum gets wet it dissolves into calcium and sulfate and may leach into the groundwater causing sulfate contamination. The U.S federal limit for sulfate in drinking water is 250 mg/L. Sometimes concentrations above this limit have been found in groundwater near unlined landfills. It also contributes to high Total Dissolved Solids (TDS) concentrations at many C&D (construction and demolition) debris landfills. [9]

Recycling is processing used materials (waste) into new products to prevent waste of potentially useful materials, reduce the consumption of fresh raw materials, reduce energy usage, reduce air pollution (from incineration) and water pollution (from land filling) by reducing the need for conventional waste disposal, and lower greenhouse gas emissions as compared to virgin production. Recycling is a key component of modern waste reduction and is the third component of the "Reduce, Reuse, and Recycle" waste hierarchy. [28]

1.2 Statement of the Problem

- Environmentally unfriendly disposal of discarded moulds in bare land which can potentially result in [9]:
  - ✓ hard water formation
  - ✓ loss of land usability
  - ✓ liable to hydrogen sulfide emission
- There are problems in relation to shortage and on-time supply of plaster of Paris. The factory is often forced to stop production. Recently the factory meets its demand from both domestic and foreign suppliers.
- Due to absence of competitive and abundant domestic suppliers, the price of virgin plaster of Paris is increasing in alarming rate. The sole public sector of calcinated gypsum producer in Ethiopia is Educational Materials and Distribution Enterprise (EMPDE) which was established in 1978 E.C. Formally established private sector producers currently are ADK, and Ehtio-gypsum which produce about 1,200 tons of gypsum annually.[16]
- Large amount of open space in the factory compound is used to accommodate the exhausted gypsum moulds starting from the establishment of the factory

1.3 Objectives of the Research

1.3.1 General Objective

The general objective of the research is to develop a process to recycle Plaster of Paris from exhausted gypsum moulds in Tabor ceramic products manufacturing share company.

1.3.2 Specific Objectives:

- To determine the most suitable temperature for regenerating plaster of Paris from exhausted and discarded moulds.
- To determine the compressive strength of the regenerated plaster of Paris.
- To determine the water absorption of the regenerated plaster of Paris.
- To determine the setting time of the regenerated plaster of Paris.
- To select and design of processes and equipments for calcining of the discarded moulds to plaster of Paris.
- To determine the proportion of the recycled and the virgin plaster to be used for best result in blending experiment.
- To determine the cacination time for best result, and composition analysis of the sample before and after calcination.
- To study techno-economic feasibility analysis of plaster of Paris recycling plant.

2. LITERATURE REVIEW

2.1 History of Gypsum

A number of examples for the utilization of plaster are reported in the literature from around 7000 to 4000 B.C, such as Catal-Huyuk in South Central Turkey. Gypsum was used in Greece in about 700 B.C. to carve statues and for various artistic applications. The technology of using the cementitious properties of gypsum was apparently obtained either from Egypt directly or via Crete. It is not clear to what extent they understood the difference between limestone and gypsum as original material or as plaster. They obviously noticed that gypsum was warm to the touch and had a higher solubility as compared to limestone, which was cold to the touch and did not suffer from solubility problems. Unfortunately, the Greeks failed to leave written records about their uses of gypsum, which is unusual since they pondered the nature of everything around them.

The modern science of gypsum began with the discoveries by Antoine Lavoisier (1743–1794) outlined in his two papers on gypsum presented to the French Academy of Sciences in 1765 and 1766. He explained that the heat treatment of gypsum resulted in the loss of about a fifth of its weight as water vapor to leave an anhydrous material. He also stated that this material when mixed with water would form gypsum again by crystallization. [23]

2.2 Types of gypsum

a. Natural gypsum

Natural gypsum consisting of mainly of calcium sulfate is a well-known common non-metallic mineral that occurs as a deposit of fairly soft and often impure rock. It was deposited from shallow seas as they evaporated about 200–300 million years ago, mostly during the Permian period. This deposition depends on pressure, temperature, and the concentration of other salts in solution. It is found in most areas near the earth surface and can be recovered relatively easily using quarries or shallow underground mines. Natural gypsum is normally white or light gray but depending on the impurities present can also be pink, dark gray, or almost black. The English term gypsum is taken from the Roman usage of the Greek “gypsos,” apparently describing a material that does not burn, although there are other interpretations.

b. Synthetic Gypsum

Synthetic gypsum is identical to natural gypsum from a chemical and crystallographic point of view. The difference between the two varieties of gypsum sources lies primarily in their physical state, which in turn depends on the origin or the manner of their formation. Synthetic gypsum is generally obtained as the final stage of industrial processes, where sulfuric acid is neutralized by a calcium salt, often $CaCO_3$. Most frequently, however, it is obtained as flue gas desulfurization (FGD) gypsum during the desulfurization of flue gasses from coal-fired power plants as FGD gypsum by the reaction of sulfur dioxide with a calcium salt. Normally calcium carbonate ($CaCO_3$) is used for this purpose or to a lesser extent calcium hydroxide ($Ca(OH)_2$). In these cases, gypsum is obtained as a filter cake in the form of wet, fine particles containing usually about 10 % water. Therefore, the difference between synthetic gypsum and natural gypsum lies initially in the original physical condition, which dictates the subsequent process steps necessary for a particular application. That is, natural gypsum exists as solid rock, which must be crushed, ground, and dried prior to its subsequent calcination. In contrast, synthetic gypsum is already finely divided but it contains more free water than natural gypsum, which must be removed by drying. Sometimes synthetic gypsum is being agglomerated to facilitate storage, transport, or further processing. It may also be ground after drying, with or without agglomeration, to change crystal shape and size distribution. [18]

2.3 Utilization of Gypsum

Natural gypsum has been used over the ages in an uncalcined form as blocks for construction purposes similar to ashlars and as the raw material to carve statues, vases, etc. More recently, utilizations of uncalcined gypsum have involved application as setting time regulator for Portland cement, as fertilizer, and for soil amelioration. Smaller amounts are used as functional filler for paints, polymers or paper.

However, most utilizations of gypsum are based on the fact that it can be calcined when subjected to heat treatment at fairly low temperatures. This results in the formation of either stucco as the first calcination stage or insoluble anhydrite as the second calcination stage.

2.3.1 Dehydration

Under atmospheric conditions, the dehydration of calcium sulfate dihydrate involves exposure to increasing temperatures with corresponding stepwise loss of water. It essentially

passes through the process indicated below. Dihydrate, hemihydrate, and insoluble anhydrite are distinct and separate phases since they have individual crystal structures .Soluble anhydrite, $CaSO_4$ (sol.) is a special case but not a separate phase. It has the same crystal structure as hemihydrate and may be considered an extension or an anhydrous variation of hemihydrate. Moreover, soluble anhydrite is unstable and reverts rapidly into hemihydrate when exposed to water vapor or when coming into contact with liquid water.

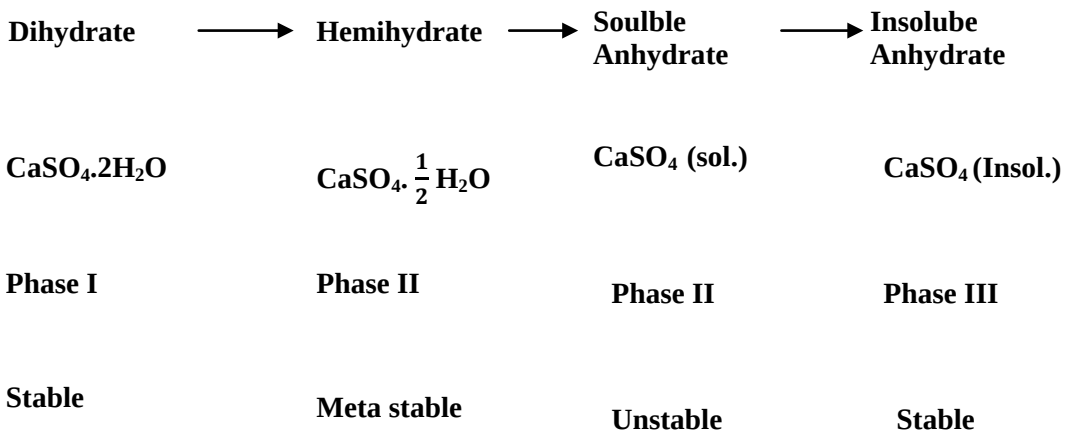

Fig.2. 1 Gypsum dehydration process (**Source:** Richard A. Kuntze 2009)

Depending upon the dehydration conditions, two variations of hemihydrate are produced, α-hemihydrate and β-hemihydrate. The α-hemihydrate is formed under normal atmospheric conditions independent of the water vapor pressure present. This is the original variety also known as stucco in practice. The more recently developed β -hemihydrate is formed in an autoclave or in salt solution between 97°C and about 160°C. This variety is mainly used for medical or dental applications to prepare molds as well as in self-leveling floor screeds. [17]

2.5.2 Rehydration

The rehydration of hemihydrate and of insoluble anhydrite to dihydrate refers to the solution of these materials in water and subsequent crystallization. This process consists of the release of heat and water which is incorporated during crystallization.

2.4 Gypsum Processing

Gypsum processing can be considered to encompass separate stages. The first consists of the preparation of the raw gypsum and covers such steps as receiving and storage, drying,

crushing, and grinding. The second stage involves the calcination of this material using a variety of equipment, such as kettles and kilns. Obviously, the essential feature of the first stage is to prepare the raw gypsum so that it is acceptable by the calcination unit. There are many procedures to accomplish this but it is always necessary to control moisture content and particle size distribution.

Natural gypsum is mined in quarries using drilling and blasting at several levels or by

underground mining using chamber blasting. This rock consists of large pieces usually containing up to 4 % free moisture. The coarse rock is conveyed to the crushing plant often located at the gypsum plant site. Impact crushers, jaw crushers, and single roll crushers with screens and oversize returns are all suitable for coarse size reduction. Impact pulverizers or roller mills are used for intermediate size reduction, and hammer mills, ball mills, and ring-roll mills are used for fine grinding. [18]

2.4.1. Receiving and Storage

Conventional gypsum processing is considered to begin when the raw material is received by the plant. Conveyors generally move rock delivered by ship to a storage site. Because natural rock is relatively coarse, rain and snow does not materially increase its moisture content. This material has usually a density of 1442 kg/m^3 and an angle of repose of 38°, which is given by the angle between the base of the material and its slope. [17]

2.4.2 Drying

If the free moisture content of the gypsum rock is below 3% it can usually bypass the dryer and move directly to the crushing stage. However, for rock with moisture content above 3% some drying may be necessary. Rock obtained from outdoor storage often contains a higher moisture content, which must be removed or reduced prior to crushing. The drying of rock can be obtained with a directly heated rotary dryer operating at 182°C. [18]

2.4.3 Crushing

Natural rock is usually reduced in size to 0.95–3.81 cm. In many plants, the rock passes through a screen and only material larger than 2.54cm is crushed. This approach allows the

use of a lower capacity mill because only about one-third of the rock is larger than 1 inch in size. Both fractions are then combined and transported by bucket or screw conveyors to a crushed rock storage bin.

2.4.4 Grinding

Crushed rock is fed into a roller mill system, which is designed to reduce the rock to an appropriate size range. As a general guide, 90%–95% passing a 100-mesh (150μm) screen is normally obtained. A variety of other mills can be used for this purpose as well. At this stage, it is possible to further reduce the moisture content of the rock by drying. [14]

2.4.5 Calcination

Calcium sulphate hemihydrate ($CaSO_4.\frac{1}{2}H_2O$) or plaster is obtained through the partial or total dehydration of gypsum at a temperature ranging from 120 to 400^oC. The structure and properties of the final product are directly dependent on the chosen Calcination condition (temperature, pressure, rapidity).Several types of calcination exist;

a. *Alpha Process* Alpha type plaster is used mainly in industrial plaster formulation for its high mechanical strength. This plaster type is the compact crystal with a low specific surface and low water demands to produce hard, low porosity casts. Alpha plaster can be formed through two different production procedures:

- **Dry process** is one that involves injection of steam vapor during calcination. The plaster is dried and treated in the regular manner.
- **Wet process** is one that involves calcination of gypsum slurry under pressure. The plaster is then spun and dried.

b. Beta process during the calcination process, under regular environmental pressure, dehydration water evaporates and a micro-porous structure is formed.

Beta plaster crystals have a high specific surface and high water demands. Beta plaster casts have porosity, but low mechanical properties and are therefore used to lightweight building application or moulds in ceramic application for their absorbent properties.

Often a mixture of both Alpha and Beta type of plaster will be used to combine the property of both and to optimize product solution to suit market requirements. [18]

2.5. Best Practice Guidelines

In order to obtain optimal result from plaster of Paris, some basic usage guidelines should be respected. Technical data sheets exist with specific usage instruction for each formula product.

2.5.1 Plaster to water ratio

It is important to respect the indicated plaster water ratio and accurately weigh the plaster and water. Variation in the plaster to water ratio have a direct influence on product characteristics: absorption capacity, density, hardness, Setting time, Sedimentation, Expansion, durability.

2.5.2 Sprinkling

Plaster is always added to water and never the contrary. It is recommended using a clean water recipient and clean tape water at constant temperature, for mixing plaster. The plaster should be sprinkled slowly and evenly across the entire surface of the water. The time taken to do this will vary between 1 and 3 minutes depending on the size of the batch. If the plaster is sprinkled quickly, dense and dry lumps can form that will not disappear during mixing. [1]

2.5.3 Soaking

The soaking time should vary between 1 and 2 minutes depending on the size of the batch. Sufficient soaking ensures that each plaster crystal is surrounded by water, and that air bubbles can escape from the mixture. If plaster is not allowed to soak for so long, air bubbles may remain in the finished plaster mould. Soaking for too long gives rise to a shorter setting time, earlier hardening and grainy surface.

2.5.4 Mixing

The mixture should be stirred with clean utensils which are free from set plaster, to obtain a homogeneous lump-free consistency. The optimum stirring time is dependent on the size of the batch and the dimension of the stirring device. If plaster is not stirred long enough it will not be evenly mixed, and if stirred too long the mixture become too thick. In both cases this has a negative effect on product characteristics. Longer stirring generally lead to great

strength, reduced absorption capacity and shorter setting time. Plaster which is vacuum-mixed has lower absorption capacity, reduced expansion and increased strength. [1]

2.5.5 De-moulding

Apply a specific lubricant or separating emulsion, in as fine a layer as possible using a brush. Wipe with cloth afterwards to avoid remnants and structuring. Incorrect or excessive use of lubricants may cause mildew, and lead to problems or defects during drying and lifting.

2.5.6 Drying plaster Moulds

Plaster moulds must be carefully dried in order to achieve even and optimum physical characteristics. Ideal drying conditions are 40-45^{o}C and 40% relative humidity for effective mould drying.

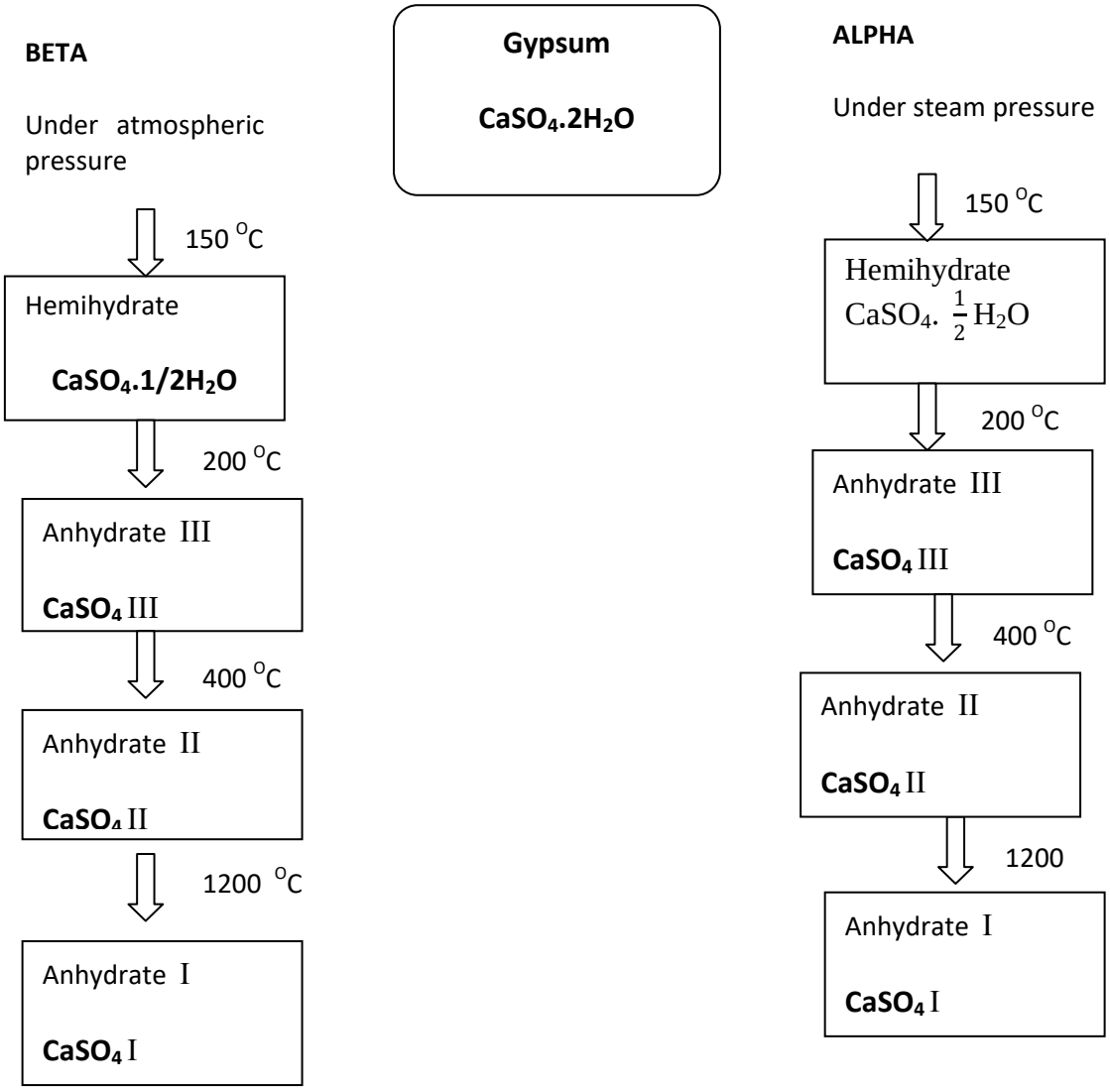

Fig.2. 2 Calcination Process (www.lafarangeprestia.com retrieved on April 5, 2014)

2.6. Calcinated gypsum in Ethiopia

Demand for calcinated gypsum in Ethiopia is being met from two sources: domestic production and imports. The domestic producers fall into three categories:

- Public sector
- Formal private sector, and
- Informal private sector

The sole public sector of calcinated gypsum producer is Educational Materials Production and Distribution Enterprise (EMPDE) which was established in 1978 E.C. with the purpose of manufacturing and selling chalk to educational institutions in the country. Although the primary purpose of its establishment was to produce writing chalk, EMPDE has also been producing and marketing gypsum, ever since it was founded, as a co-product.

The maximum attainable capacity of EMPDE for gypsum production is 1,300 tons per annum, while during years 1990 – 1996 E.C, production of gypsum by the Enterprise varies from 314.3 tons in 1990 to 744.2 tons in 1996.

Table 2.1: Production of gypsum by EMPDE (1990-1996 E.C.)

Year (E.C)	Production (tons)
1990	314.3
1991	408.1
1992	479.1
1993	438.5
1994	550.2
1995	459.8
1996	744.2

Source: Customs Authority, External Trade Statistics, Annual Issues.

In estimating the 1999 production level of EMPDE it is assumed that the last three years in the data set (1994 – 1996) average annual production (585 tons) approximates production level of the enterprise.

Formally established private sector producers currently are ADK, and Ehtio-gypsum which produce about 1,200 tons of gypsum annually.

There are also small informal sector producers who do not have standard plant and machinery but rather carry out the process of production using crude makeshift methods. Such cottage producers' number is about 5, and the combined supply of calcinated gypsum originating from the informal sector is estimated at 400 tons per annum.

Although the bulk of demand for calcinated gypsum is met through local production, some amount is also imported for various purposes. The gypsum imported from overseas is generally of a higher quality and fineness; and is mainly used for medical (bone casting), ornamental and industrial purposes.

The import data of gypsum during a ten years period of time (1997 - 2006) is shown in Table2.2

Table 2 2: Import of calcinated gypsum (1997 – 2006)

Year, GC	Quantity Imported (tons)
1997	60.7
1998	17.2
1999	20.2
2000	88.4
2001	132
2002	163.7
2003	76.4
2004	4.93
2005	5.48
2006	109.63
Average	**65.86**

Source: Customs Authority, External Trade Statistics, Annual Issues.

As can be seen from Table 2.2, import of calcinated gypsum shows a highly fluctuating trend form year to year. However, during the period of analyses the average annual import was 65.86 tons, which is assumed to indicate the present level of import.

Aggregating all the above source of supply to the local market of gypsum, the present total supply of the product is thus estimated at 2,254 tons. Assuming supply is in equilibrium with demand, this figure could fairly approximate present effective demand

Projected Demand

The demand for gypsum, like many other construction materials, is a function of a number of interrelated variables. These variables that are essential in determining the magnitude and trend of the demand for gypsum are:

- The overall economic development level and growth trend of the country,
- The pattern and trend of the construction industry in general and the building construction sector in particular,
- Expected technological change that affects the structure of the construction industry,
- Government policies and regulations that have impact on the future level and trend of construction activities, and
- Size of population and its growth rate.

Table 2.3: Projected demand for calcinated gypsum

Year GC	Projected Demand (tons)	Existing Local Supply (tons)	Demand Gap (tons)
2008	2412	2185	227
2009	2581	2185	396
2010	2761	2185	576
2011	2955	2185	770
2012	3161	2185	976
2013	3383	2185	1198
2014	3619	2185	1434
2015	3873	2185	1688
2016	4144	2185	1959
2017	4434	2185	2249
2018	4744	2185	2559
2019	5076	2185	2891

2020	5432	2185	3247
2021	5812	2185	3627
2022	6219	2185	4034

Source: Customs Authority, External Trade Statistics, Annual Issues

2.7 The ceramic industry

Generally the term “ceramics” (ceramic products) is used for inorganic materials (with possibly some organic content), made up of non metallic compounds and made permanent by firing process. In addition to clay based materials today ceramics include a multitude of products with a small fraction of clay or none at all. Ceramics could be glazed or unglazed, porous or vitrified. [8]

Firing of ceramic bodies induces time -temperature transformation of constituent materials usually in to a mixture of new materials and glassy phases. Characteristic property of ceramic products include high strength, wear resistance, long service life, chemical inertness and non toxicity, resistance to heat and fire, (usually) electrical resistance and sometimes also a specific porosity.

Tabor ceramics products manufacturing share company was established in 1980 in Hawassa city with a design capacity of 6000 tons per year. The factory constitutes three production lines; sanitary wares line (1000 tons/year), table wares line (2000 tons/year), tiles (3000 tons/year).

The main reason of the factory being established in Hawassa is proximity to the source of most of the raw materials like Kenticha (feld spar and quartz), Bomboha (bomboha kaoline), Hossana (Hossana kaoline), Muger (mugger clay), Derba(silca sand and limestone).Besides to these, the factory imports not more than 10% of the inputs from abroad; ball clay and frit glaze (from China and India). About half the production cost is incurred for fuel in the 3 production line kilns for firing.

2.7.1 Process description

2.7.1.1 Body preparation

The basic raw materials needed for sanitary wares are: hosanna kaoline, ball clay, feld spar and silica sand. Out of which the ball clay is the imported ingredient for its high plasticity. The proportion is not fixed, but in general 50% plastic materials (clays) and 50% hard materials (feld spar and sand) are used in a single charge. Recently the following amount is used at a time:

The clay ingredients give plasticity to enable the body to be shaped and give it adequate strength so that the wares could be handled safely between the shaping and the firing process. The feldspar melts on the firing and reacts with the body constituent to form a glass and strengthen the fired product. Silica in form of sand added to body control the thermal expansion of the body.

Table 2.4: Body composition per charge for sanitary wares

Ingredient	Composition (%)	Wt(Kg)
Feldspar	23.8	1440
Silica sand	25.8	1560
Hosanna kaoline	31.1	1880
Ball clay	20.7	1250

Source: Tabor ceramic products manufacturing share company

a. Milling Once the raw materials are prepared in the correct proportion, it is charged into a ball mill (except the ball clay) where crushing by the centrifugal action of the rotating mill and the balls take place. The mill is cylindrical in shape and is made of stainless steel. Both the internal lining and the balls are made of ceramic making materials to avoid product contamination. The mill is made to rotate with pre-calculated rotational speed to (15 rpm) for 10-12 hours until the required particle size is obtained. The age of the balls affect the process as the balls wear through time and get less efficient in milling. The particle size is the controlling parameter for proper milling. Sample from the mill is taken and particle size is measured using hydrometer method which is based on the principle that coarse particle settle faster under the action of gravity.

b. Blunging

If the milling is acceptable, the mixture from the mill is emptied by the pipeline into the first blunger. Only opening the lid of the mill is enough to discharge the content by atmospheric pressure. There are five blungers in the body preparation section (A, B, C, D, E).The function of the blungers is mixing and uniforming the body composition. Ball clay is added usually in blunger-A together with some scrap to the virgin slip. Deflocculates like sodium silicate (12-15 kg per batch) is added to get homogenous and deflocculated slip. Sample is taken from the slip and the density, thixotropy, and fluidity are measured. The fluidity of the slip is the affinity to flow and the thixotropy of the slip is the density difference between two time intervals. At this stage, the density should be 1.845-1.865 kg/L, the fluidity 260-290 degree, and the thixotropy should be 45-60 degree for the slip to be acceptable.

c. Sieving and magnetic separation

The slip from the blunger is directly transported to the vibrating sieves using compressed diaphragm pumps. The sieves are two in number and they work in turn, while one is working the other is cleaned. The sieves separate coarse particles from the slip which are then dried and re-milled latter. On bottom of the vibrating sieves lie permanent magnets which separate iron and other magnetic materials which otherwise will produce spots and coloring problem upon firing. The fired slip is collected and stirred in two blungers D-1 and D-2 until it is transported to the casting room slip storage tanks H-1 and H-2. The slip is left to age before casting for about 24 hours.

d. Mould Making

The mould which is used for casting process in the ceramic industries is prepared by mixing plaster of Paris and water in the correct proportion. Plaster moulds are a good bed for cake formation due to their high strength and water absorption; on the other hand the quality of final product is affected directly by the quality of plaster mould. Furthermore, the quality of plaster mould can control casting waste and quality of final product in sanitary ware industry . While plaster mould do its important role which is water absorption by their capillary volumes, plaster mould must have proper strength and accurate dimension, they also must have smooth surface by eliminating bubbles and cavities.[12]

$$CaSO_4.\frac{1}{2}\,H_2O \quad + \quad \frac{3}{2}\,H_2O \longrightarrow CaSO_4.2H_2O$$

Plaster Water Gypsum (Mould)

Fig.2. 3 Gypsum moulds

2.7.1.2 Casting

Forming of sanitary ware items is performed in gypsum moulds; moulds are prepared from plaster of Paris which is calcium sulphate hemihydrates, $CaSO_4.\frac{1}{2}\ H_2O$.

After the slip has undergone the necessary aging step in the body preparation room, it is transported by compressed air diaphragm pump through pipes and stored in the casting room H-1 and H-2 tanks. From these tanks the slip is transported to the casting benches.

Casting procedure:

- Clean dirt and clay from the moulds by dry brush and air hose.
- Dust the mould by talcum powder to avoid sticking.
- Assembling the mould parts tightly with bonder or bolt and nut system.
- Adjusting filling tubes and closing draining holes of the mould by wood.
- Pouring the slip slowly into the mould and top up it when the slip gets sucked in.
-

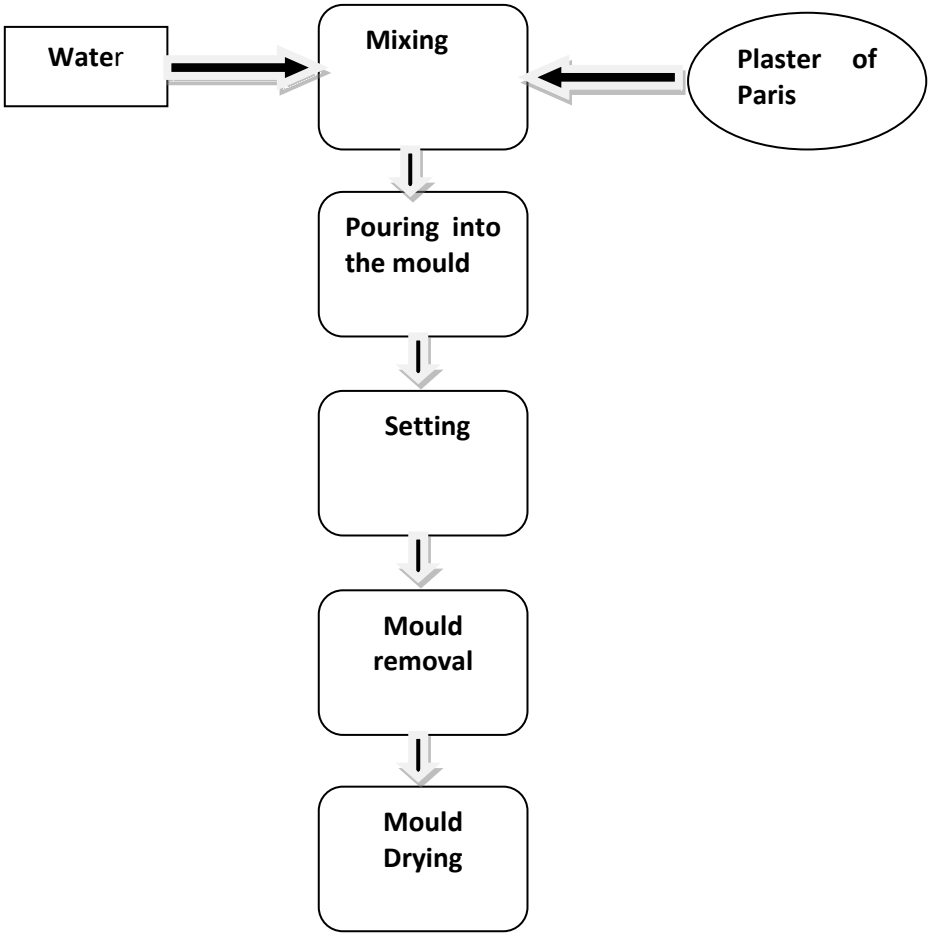

Fig.2. 4 Mould Making

There are two types of casting for sanitary wares. The first one is open casting where the slip is poured into the mould which has inside opening similar to the required item. Cistern tank is made by this type of casting, here once the mould is properly filled with the slip, it absorbs the water within and yields the required item. There is no slip to be drained off after sometime.

The second and more practiced casting of sanitary wares in the room is drain casting where the slip is charged into the mould, waited until it absorbs some water and makes some layer and excess slip is drained off.

The casting time is mainly a function of the slip density and the mould age. A less dense slip takes more time to thick and relatively old mould or mould made from impure plaster has lower water absorbing efficiency. Mostly the casting time is 45 to 60 minutes. After this the excess slip is drained off using containers for bottom discharge. The drained slip is stored in blunger E, available in the casting room until it is pumped back to the body preparation room. After drainage the formed item is made to dry using the room heated air circulated by ceiling fans so that it will be strong enough to take it out of the mould. For some items like bottom outlet parts are casted separately are assembled at this stage. Then pours are made, cracks maintained and surface smoothening and finishing works are made here before drying.

2.7.1.3 Drying

Drying is carried out before firing to give the products enough strength to be handled safely for firing and also to remove the risk of wares being shattered in the kiln by rapid evolution of steam. The amount of water which has to be removed depends on the making technique employed. For sanitary wares which are made by slip casting about 18-20% water has to be removed. The casting room serves as the first drying place before items are dried in the main drying room.

After the casting is done, hot air generated by exchanging hot air from the boiler room and air sucked in by fans is used to dry items casted. The drying process is performed slowly by covering items using plastic to avoid cracking by rapid drying of the surface and wet interior. Final drying takes place in the main drying room after the semi-dried items are fettled and selected in the casting room.

2.7.1.4 Glazing

A glaze is a glass. It is a vitreous material which in effect is a super-cooled liquid which has cooled below the point it might have crystallized. It is liquid with very high viscosity. There are several for applying glaze on the surface of the product. [8]

- It provides an impermeable surface to what otherwise be an open porous product. This surface is hygienic and easily cleaned.
- It improves the appearance of the product by giving it smooth glassy surface.
- It provides a surface for carrying decoration applied on the surface or provides protection to decoration applied under the glazed surface.

- It increases to some extent the strength of the ware.

Sanitary ware glaze is prepared by mixing 15% opaque glaze and 85% local glaze, 10% zirconium sulphate.

Local glaze ingredients: Feldspar= 41%,

Quartz=33%,

Bamboha kaolin=10%,

Limestone=16%

Glaze is applied by manual spraying on the dried and inspected products.

2.7.1.5 Firing

Firing converts the weak dried green wares into strong articles of low or zero porosity by formation of a glassy phase which flows into the pores between particles and solidifies on cooling. Firing and re-firing of sanitary wares is carried out in one time firing shuttle kiln. Kilns are basically two in types, intermittent (shuttle) kiln and continuous (tunnel) kiln. In the firing of shuttle kiln, wares are placed in the kiln, the temperature is gradually raised, wares soaked in at constant temperature, the kiln is then cooled and wares are then removed and replaced by ware for firing.

The main advantage of shuttle kiln over the tunnel kiln is it is flexible. Firing can be carried out at any temperature required or the kiln can be closed down and no firing at all.

Firing ignited using fuel (kerosene) and the combustion air is atomized towards the items. There are 10 burners 5 at each side. The maximum temperature worked with recently is 1220 ^{o}C. The items to be fired are soaked for one hour at this maximum temperature. The firing temperature reaches this maximum point after 12 hours and 15 minutes of ignition. Then the fired items are cooled for the next 9 hours and 30 minutes. The stack gas is directed to the main chimney by the holes available on the roof of the kiln. The flue gas exhaust system is natural draught equipment, the draught energy is provided by the temperature gradient between the hot waste gas and the cooled ambient air as well as the chimney height. High quality hard and light bricks with ceramic fiber are used for lining and insulating. Side

useful volume of the kiln for firing is 41.12m^3 from the five kiln cars for firing about 4.5 tons items in a cycle. The doors of the kiln are operated using hydraulic system.

2.7.1.6 Inspection

After firing items that satisfy the quality requirement and those that need repair firing are selected. Products with little defects are re-glazed and maintained using ball clay and re-fired before packing and storage.

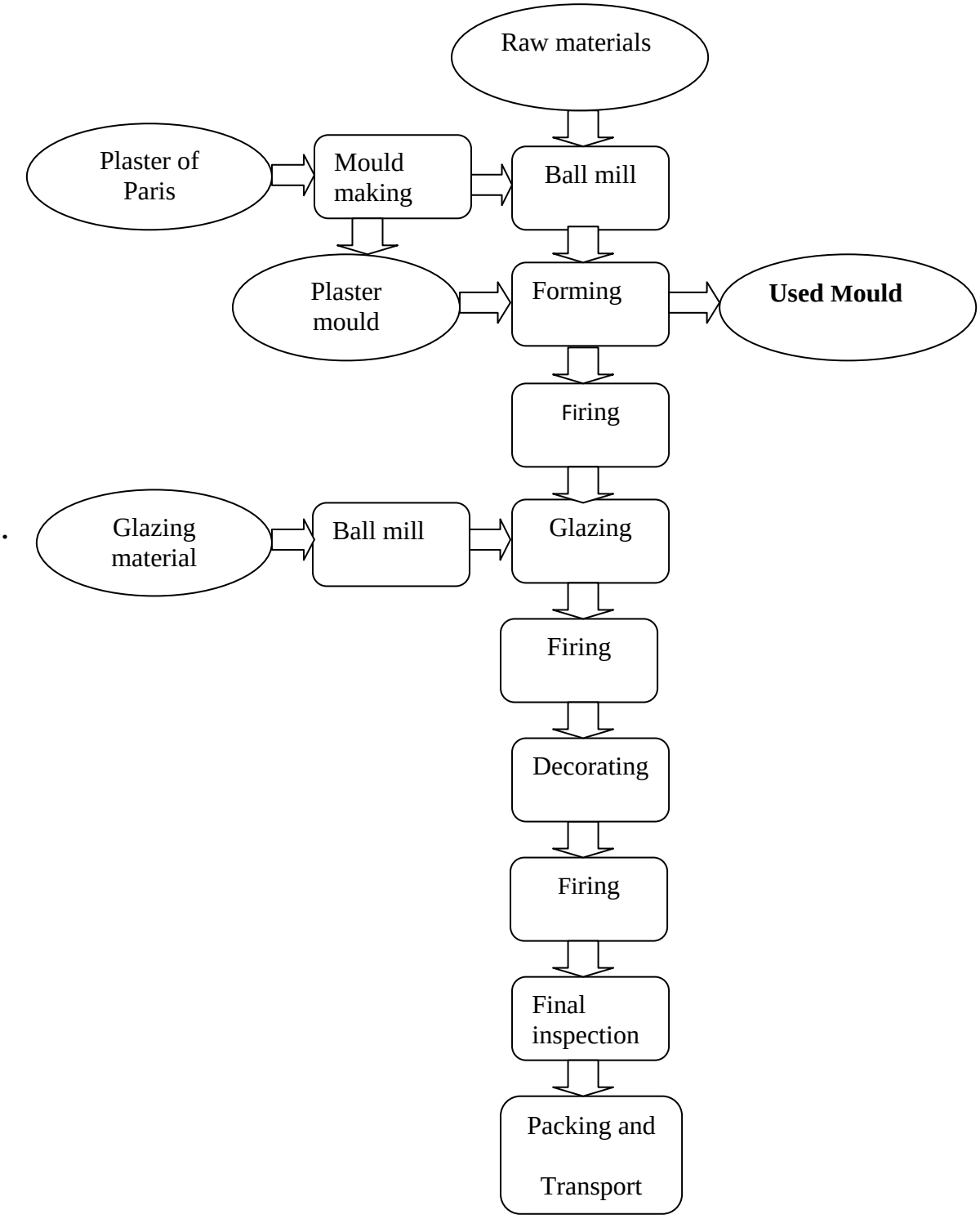
Raw materials
Plaster of Paris
Mould making
Ball mill
Plaster mould
Forming
Used Mould
Firing
Glazing material
Ball mill
Glazing
Firing
Decorating
Firing
Final inspection
Packing and Transport

Fig.2. 5 Discarded Plaster Moulds

3. MATERIALS AND METOHDS

3.1 Materials and Equipments

The major raw materials used during the experiment are discarded gypsum mould, virgin plaster of Paris, surfactant mould release, wax, and pure water. The discarded gypsum mould was brought from Tabor ceramics products manufacturing share company after it had been used exhaustively for casting.

The equipments used during the experimentations are laboratory ball mill, oven, sieves of different mesh size, mixing bowl, hand stirrer, compressive strength tester machine, balance, Petri dish, rod casting plates and drying trays.

3.2 Experimental Methods

3.2.1 Raw Material Cleaning

First the discarded moulds are cleaned of clay, sand, dust and deflocculated crystals deposited on the mould surface, the moulds are then crushed and the fine lumps are washed with water, which will remove the soluble deflocculates. The material is then dried for further processing.

Fig.3. 1 Discarded gypsum mould

3.2.2. Crushing

The cleaned gypsum mould is crushed to 25-100 mesh size. First it is broken to smaller pieces manually in the varying sizes of up to 150 mm. These lumps are crushed to uniform sizes in the range of 25-100 mesh in the crusher. The crushing is carried out in ball mill. A ball mill, a type of grinder, is a cylindrical device used in grinding (or mixing) materials like ores, chemicals, ceramic raw materials and paints. Ball mills rotate around a horizontal axis, partially filled with the material to be ground plus the grinding medium. [23] Ceramic balls were used as a grinding media.

Fig.3. 2 Laboratory ball mill

3.2.3 Calcination

The process of calcination to form plaster of Paris is merely one involving heating of the cleaned, dried, and crushed gypsum to a certain temperature to drive off a portion of the water of crystallization. In order to obtain uniform results and produce a good quality plaster of Paris from the discarded moulds, it is highly desirable that the temperature should be evenly distributed to the particles of the gypsum so that every particle will be heated to the temperature necessary for calcination and no parts of the gypsum shall be heated too highly.[26] Therefore Calcination of the gypsum powder is conducted at different temperature and to make the temperature even throughout mixing is conducted with certain time interval. The beta type of calcination is used where water evaporates and a micro-porous structure is formed under regular environmental pressure. The calcination experiment is conducted in the laboratory dryer.

Fig.3. 3 Calcination process in laboratory dryer

3.2.4 Grinding

The crushed and calcined mould is once again fed into a ball mill system, which is used to further reduce the mould to an appropriate size range size of 150-250 µm. sieving and re-milling is conducted until the appropriate particle size is maintained. Crushing is performed by the centrifugal action of the balls in the rotating cylinder.

3.2.5 Casting Plaster Rods

3.2.5.1 Preparing the Case Mould

First the pre-prepared wooden case moulds for casting are rinsed with liquid soap as surfactant and shake dry to avoid sticking of the casted rod in de-moulding. The table where the slip is poured is made level.

Fig.3. 4 Wooden case mould

3.2.5.2 Adding Water to the Mixing Bowl

Flexible poly ethylene easy for cleaning is used as mixing bowl. Cold water is used as it gives a little extra time to work before the plaster sets up

3.2.5.3 Adding the Plaster

Plaster is added to the water and never water to the plaster. The plaster water ratio used for mixing is 100:75.Then the plaster is swift slowly into the water until all the available water is filled with plaster. Now the container is left untouched for 1 to 2 minutes while the plaster absorbs the water.

Fig.3. 5 Adding plaster to the water

3.2.5.4 Mixing the Plaster

Using a flat bladed plastic bowl scraper, hand mixing is done slowly to avoid beating air into the mix. Mixing continues until a smooth creamy mixture is formed. Over mixing will cause the plaster to set up quicker often before it can be poured into the moulds so the mixing is done only for 30 seconds. Lumps formed on the process are removed.

3.2.5.5 Filling the Moulds

The plaster is poured slowly into the moulds until full. over filled and excess plaster was removed by wiping over the surface with a flat bladed paint scraper. The moulds is then left untouched for the plaster to harden.

3.2.5.6 Removing the Plaster Casts

Finally the plaster cast is removed slowly from the mould to remove damage. There is liberation of heat that can be sensed by hand touching when the plaster fully sets, this time the cast can be de-moulded.

3.2.5.6 Drying the Plaster Casts

Plaster moulds must be carefully dried in order to achieve even and optimum physical characteristics. The casted test bars are dried at a temperature of 45 ^{o}c for 24 hours in the laboratory drier.

Fig.3. 6 Drying plasters rods

3.2.6 Setting Time Determination

Plaster is added to the water and sifted through spoon to spread evenly over the surface of the water. The plaster/ water ratio used in the experiment was 100:75 over a period of 30 second. The mix is allowed to soak for a further 30 seconds and then stirred by hand using spoon until it starts to thicken and be ready to pour into the wooden moulds to give the test specimen. The blending time is the time interval from the end of the initial 30 second soaking period to the moment when the thickened plaster slurry is poured. The setting time is taken from the time plaster slurry is poured in to the moulds until it has set hard to the touch by hand. Upon setting there is liberation of heat which can be well felt by touching.

Fig.3. 7 Casted rod

3.2.7 Determination of Water of Absorption

The samples are well dried for period of 24 hours at a temperature of 45^{o}c and cooled before the water absorption test is performed. The method used here for measuring the water absorption is the soaking method where water is filled in the soaking dish for height of 4mm [24].The plaster rods are marked at different height and specifically for this purpose at height of 6 cm. The sides of the plaster rod immersed in the water are covered by transparent plaster so that absorption takes place only through the end of the plaster rod. The time is taken immediately when the rods are immersed into the soaking dish till it crosses the 6cm mark on one of the sides. Then the water absorption affinity is calculated by dividing the total height travelled by the time taken.

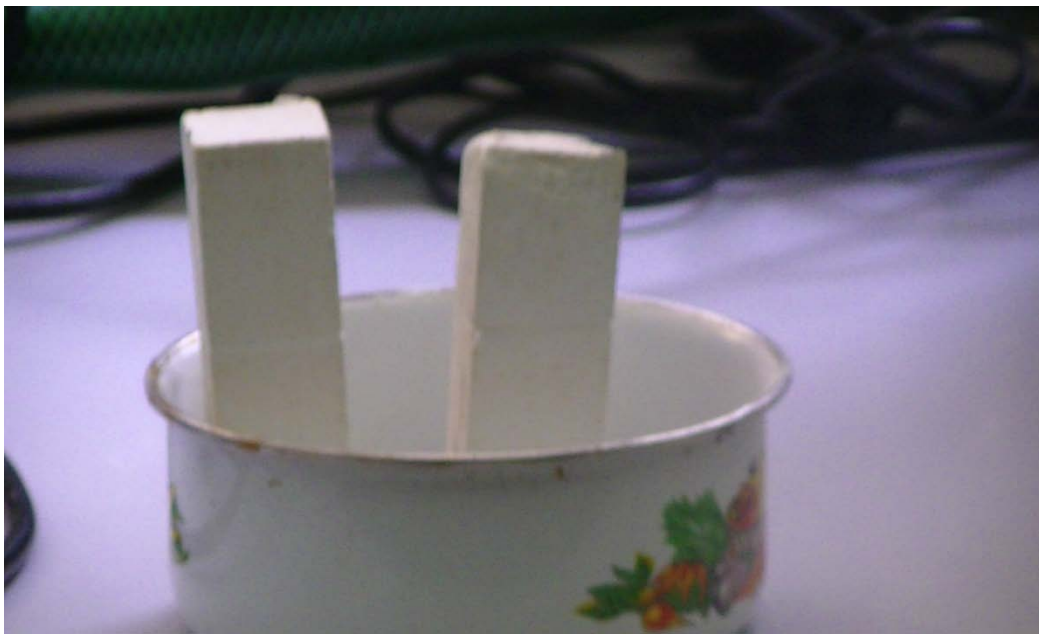

Fig.3. 8 Water absorption test

3.2.8 Compressive Strength Determination

By definition, the compressive strength of a material is that value of uniaxial compressive stress reached when the material fails completely. The compressive strength is usually

obtained experimentally by means of a compressive test. The apparatus used for this experiment is the same as that used in a tensile test. However, rather than applying a uniaxial tensile load, a uniaxial compressive load is applied. As can be imagined, the specimen (usually cylindrical) is shortened as well as spread laterally.

Compressive strength analyses were conducted on the samples using compressive strength testing machine available in civil engineering department of AAIT.

Fig.3. 9 Compressive strength testing

3.2.9 Determination of Mole of Water of Crystallization

In crystallography, water of crystallization or water of hydration or crystallization water is water that occurs in crystals. Water of crystallization is necessary for the maintenance of crystalline properties, but capable of being removed by sufficient heat. It is the total weight of water retained by certain salts at a given temperature and is mostly present in a definite (stoichiometric) ratio. [11]

The mole of water of crystallization present was determined using gravimetric analysis. the sample powder was placed in the Petri dish and oven-dried for six hours at 180^{o}C

after being weighed by balance. This enables the determination of mass of anhydrous calcium with which were determined the moles of water given off and that of the bone-dried calcium sulphate. The water of crystallization was determined as the quotient of the moles of water lost over moles of anhydrous $CaSO_4$

Mass of Dish, Md
Mass of Dish + Mass of powder, Mdp
After heating for six hours at 180^0C
Mass of Dish + Mass of dried Powder, Mdd
Mass of water lost, Mw=Mdp-Mdd
Molar mass of water = 18 g/mol
Mole of water lost, Mow =Mw /18
Mass of anhydrous $CaSO_4$=Mad = Mdd – Md
Molar mass of CaSO4 = 136.15 g/mol
Mole of anhydrous $CaSO_4$ = Mad/136.15
To determine x in $CaSO_4 .xH_2O$ (i.e., ratio of mole of water to $CaSO_4$):
x = Mole of water lost /Mole of anhydrous $CaSO_4$
The mole of the water of crystallization is x should be close to 0.5 for the recycled and 2 for the discarded moulds.

3.3 Experimental Design for Recycling Plaster of Pairs

In this work recycling of plaster of pairs from discarded gypsum moulds is performed using exhausted moulds brought from Tabor ceramics products manufacturing share company. Experimental design was analyzed and done by the Design-Expert 7.0.0 program.

The experimental design selected for this study is Central Composite Design (CCD) and the responses measured are setting time, water absorption, and compressive strength.

The process variables studied are calcination temperature, and calcination time. A five-level-two-factor CCD was employed in the optimization study, requiring 21 experiments. The calcination temperature and calcination time were the independent variables selected to optimize the setting time, water absorption, and the compressive strength.

The 21 experiments were carried out and data was statistically analyzed by the Design-Expert program to find the suitable model for determining the response variables.

.

Table 3.2 shows the complete experimental design matrix of CCD for the factorial design. The order in which the runs were made was randomized to avoid systematic errors.

Table 3. 1: Independent variables and levels used in the central composite design for recycling

Variables (factors)	Factor Coding	Unit	Levels				
			-2	-1	0	+1	+2
Temperature	X1	^{o}C	140	141.56	145	148.54	150
Time	X2	Hour	2	2.15	2.5	2.85	3

Table 3. 2: Central Composite Design Arrangement (Experimental Design Matrix)

Run	Temperature, ^{o}C	Time, Hr	Setting time, min	Water absorption, cm/min	Compressive strength, Mpa
1	145	2.50			
2	141.46	2.85			
3	141.46	2.15			
4	148.54	2.15			
5	145	2.5			
6	145	2.00			
7	150	2.50			
8	145	2.00			
9	145	2.50			
10	140	2.50			
11	145	3.00			
12	145	2.50			
13	150	2.50			
14	141.46	2.85			
15	141.46	2.15			
16	148.54	2.85			
17	148.54	2.85			
18	145	3.00			
19	148.54	2.15			
20	145	2.50			
21	140	2.50			

4. RESULT AND DISCUSSION

4.1 Impurity content, Water of crystallization and Color of Mould

4.1.1 Impurity Content Determination

500 gm dried gypsum was broken into smaller lumps and washed with water to remove clay, dust and soluble deflocculates during casting. Then it was dried, crushed and sieved with 250 µm sieve. After cleaning, 458 gm of gypsum was obtained.

Hence % of impurity = $\frac{\text{wt.before cleaning} - \text{wt.after cleaning}}{\text{wt.before cleaning}} * 100$ ……4.1

$$= \frac{500\,\text{gm} - 458\,\text{g}}{500\,\text{g}} * 100$$

$$= \frac{42g}{500g} * 100$$

$$= 8.4\%$$

4.1.2 Determination of Mole of Water of Crystallization

a. Water of crystallization of the discarded mould powder

Mass of Dish, Md = 42.3g

Mass of Dish + mass of powder, Mdp = 62.3g

After heating for six hours at 180^0C

Mass of Dish + Dried Powder, Mdd = 58.2g

Mass of water lost, Mw=Mdp-Mdd =4.1g

Molar mass of water = 18 g/mol

Mole of water lost, Mow =4.1 /18 =0.227 mol

Mass of anhydrous CaSO4=Mad = Mdd – Md =58.2g-42.3g=15.9g

Molar mass of CaSO4 = 136.15 g/mol

Mole of anhydrous CaSO4 = 15.9g/(136.15g/mol) =0.116 mol

To determine x in CaSO4 .xH2O (i.e., ratio of mole of water to CaSO4):

x =0.227/0.116=1.943

x ≈2

Therefore, the molecular formula of the compound is: $CaSO_4 . 2H_2O$

b. Water of crystallization of the recycled powder

Water of crystallization of the recycled mould powder

Mass of Dish, Md = 42.3g

Mass of Dish + recycled powder, Mdp = 62.3g

After heating for six hours at 180 ^{o}C

Mass of Dish + Dried Powder, Mdd = 61.16g

Mass of water lost, Mw=Mdp-Mdd =1.14g

Molar mass of water = 18 g/mol

Mole of water lost, Mow =1.14g /(18g/mol) =0.063 mol

Temperature, ^{o}C	Observed color

Mass of anhydrous $CaSO_4$=Mad = Mdd – Md

61.16g-42.3g=18.86g

Molar mass of $CaSO_4$ = 136.15g/mol

Mole of anhydrous $CaSO_4$ = 18.86g/ (136.15g/mol) =0.138mol

To determine x in $CaSO_4 .xH_2O$ (i.e., ratio of mole of water to $CaSO_4$)

x =0.063/0.138=0.456

x ≈0.5

Therefore, the molecular formula of the recycled powder is: $CaSO_4.0.5H_2O$, hence there is reversion of the mould from $CaSO_4.2H_2O$ to $CaSO_4.\frac{1}{2}\ H_2O$

4.1.3 Color of Mold

The raw Plaster of Paris has crystal white color [12]. The recycled powder regained whitish color with rise in calcination temperature as seen in Table 4.1. The calcination at 100^{o}C gave brownish-white coloration to the powder when mixed with water. The appearance improved with increase in temperature to 120^{o}C which gave less-brownish white to the powder-water mixture. At 140^{o}C, the recycled powder regained the quality white color of the original substance giving white coloration to water-powder mixture.

The recovery of the whiteness of the recycled powder as the temperature rises is a good indication of process reversal from dihydrate form to the hemi-hydrate form.

Table 4. 1: Observed colors of the recycled POP at different calcination temperatures

100	Brownish white
120	Less brownish white
140	White
160	White

Fig.4. 1 Color of the Powder before and after Calcination

4.2 Analysis on Gypsum Mould Recycling

The experimental design selected for this study is Central Composite Design (CCD) and the responses measured are setting time, water absorption, and compressive strength. The two input variables are the calcination temperature and the calcination time.

The Design-Expert 7.0.0 program was used in the regression analysis and analysis of variance (ANOVA). The Statistical software program was used to generate surface plots, using the fitted equation obtained from the regression analysis.

The parameters with their respective working ranges in the factory and the central composite design conditions and responses are given in Tables 4.2 and 4.3 respectively.

Table 4.2: Parameters and working ranges for Tabor Ceramic Products manufacturing share company

Parameter	Unit	Working range
Setting time	minute	15-20
Water absorption	cm/minute	0.15-0.35
Compressive strength	MPa	≥5.0

Source: Tabor ceramic Products Manufacturing Factory

Table 4.3: Experimental Design Result

Run	Temperature, ^{o}C	Time, hr	Setting time, minute	Water absorption, cm/min	Compressive strength, MPa
1	145	2.50	16.5	0.29	5.4464
2	141.46	2.85	18.5	0.32	5.4998
3	141.46	2.15	15	0.33	5.1169
4	148.54	2.15	14.6	0.25	5.7010
5	145	2.5	16.3	0.29	5.4664
6	145	2.00	13.9	0.28	5.3850

7	150	2.50	16.4	0.24	5.6850
8	145	2.00	14	0.29	5.3799
9	145	2.50	16.6	0.28	5.4655
10	140	2.50	16.8	0.34	5.2451
11	145	3.00	19.2	0.28	5.5451
12	145	2.50	16.6	0.29	5.4659
13	150	2.50	16.5	0.24	5.6849
14	141.46	2.85	18.5	0.31	5.4992
15	141.46	2.15	14.9	0.32	4.9886
16	148.54	2.85	18.3	0.25	5.3682
17	148.54	2.85	18.4	0.25	5.3672
18	145	3.00	19.3	0.28	5.5449
19	148.54	2.15	14.8	0.24	5.6870
20	145	2.50	16.6	0.28	5.4227
21	140	2.50	16.8	0.34	5.1987

4.2.1 Statistical Analysis on Setting Time

From the over view of the result (Table 4.3), the maximum setting time is 19.3 minutes, at a calcination temperature of 145^{o}C and calcination time of 3 hours. At this point, the respective water absorption and compressive strength test values are respectively 0.28cm/min and 5.5449MPa which are both well within the acceptable range of the factory. The minimum setting time is 14 minutes which is the lower allowable limit for casting. Setting time value below the lower limit means the plaster slip hardens fast before getting the appropriate shape in the case mould. Setting time values higher than the upper limit results in low production rate of the plaster moulds for casting purpose.

The setting time values were used to develop a mathematical model that correlates the setting time to the calcination temperature and calcination time. Design Expert software version 7.0.0 was used for the regression analysis of the experimental data and also for evaluation of the statistical significance of the equation developed.

The model was tested for adequacy by analysis of variance. The regression model was found to be highly significant with the correlation coefficients of determination of R-Squared (R^2), adjusted R-Squared and predicted R-Squared having a value of 0.9967, 0.9963 and 0.9958, respectively.

Based on the analysis of variance, the setting time was not affected by interaction effects of the process variables. The factor that affect significantly the setting time is the calcination time. Figure 4.4 shows the effect of calcination time on setting time. It can be seen that with increasing calcination time the setting time increases significantly. The effect of calcination temperature on setting time is not significant and the result is shown in figure 4.3

Table 4.4: Analysis of Variance (ANOVA) for setting time

Source	Sum of square	Df	Mean square	F value	p-value prob> F	Remark
Model	54.19	2	27.09	2693.20	<0.0001	Significant
A-temp	0.20	1	0.20	19.90	*0.0003*	
B-time	53.99	1	53.99	5366.50	*<0.0001*	
Residual	0.18	18	0.010			
Lack of fit	0.068	6	0.011	1.21	0.3675	Not significant
Pure error	0.11	12	9.417E-003			

Cor Total	54.37	20				

The Model F-value of 2693.20 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant.
In this case A, B are significant model terms. Values greater than 0.1000 indicate the model terms are not significant
The "Lack of Fit F-value" of 1.21 implies the Lack of Fit is not significant relative to the pure error. There is a 36.75% chance that a "Lack of Fit F-value" this large could occur due to noise.

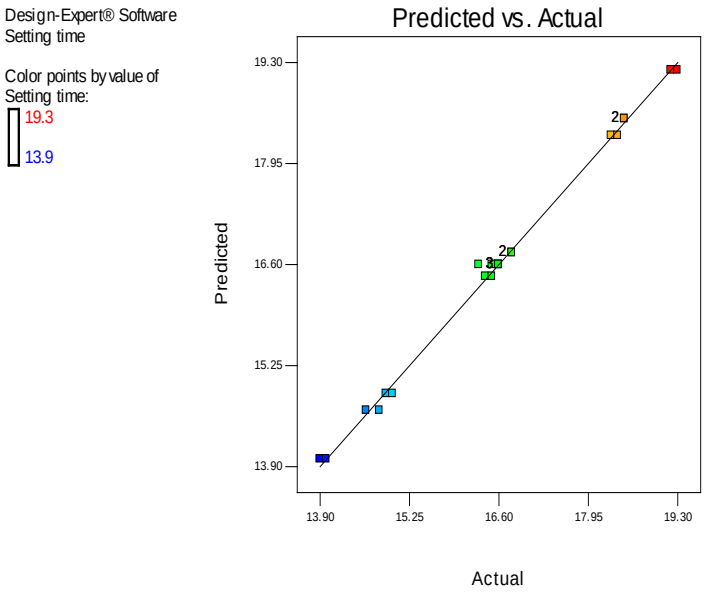

Fig.4. 2 Predicted versus Actual Setting time

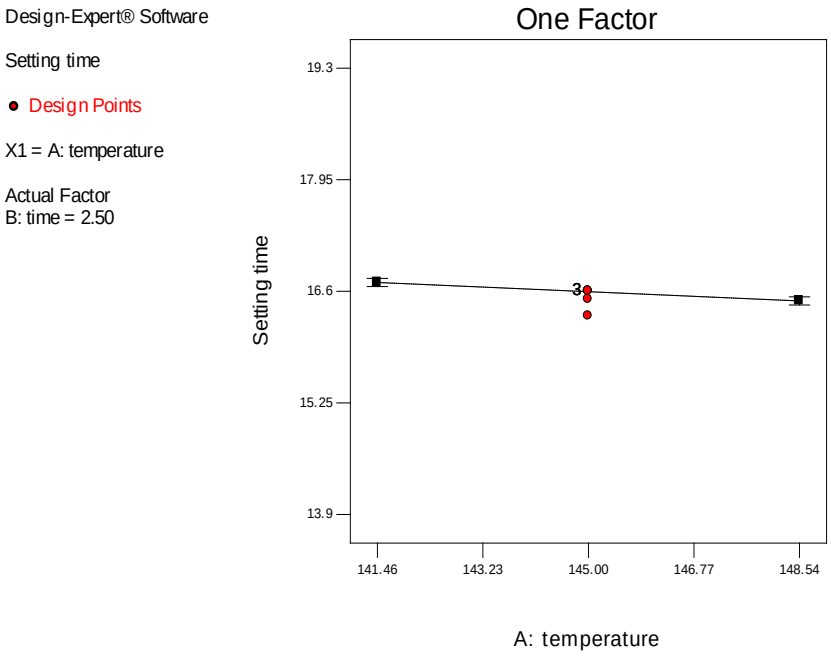

Fig.4. 3 Effect of Calcination temperature on setting time

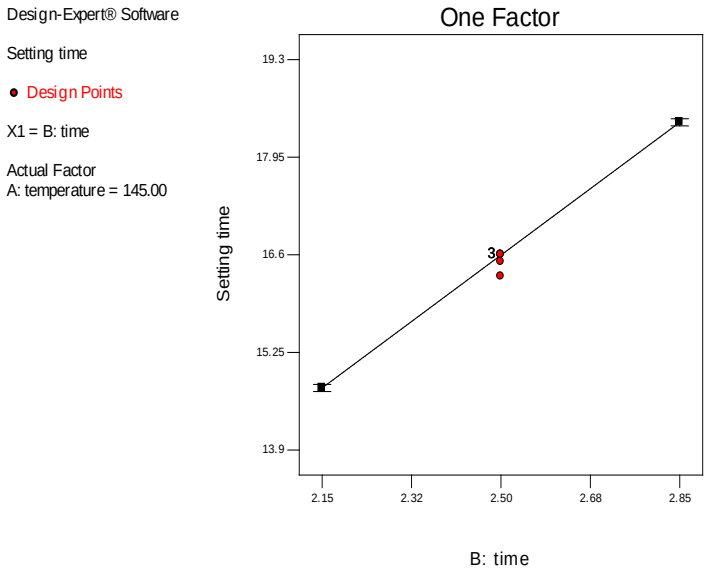

Fig.4. 4 Effect of Calcination time on setting time

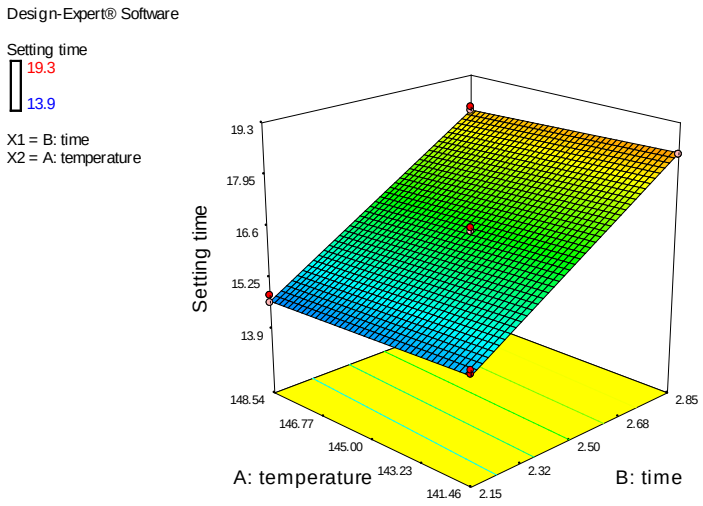

Fig.4. 5 Surface plot of the effect of Calcination temperature and Calcination time on Setting Time

Development of the Regression Model

The model equation that correlates the response (setting time) to the calcination process variables, calcination temperature and calcination time is:

Setting time (min) = 8.1943 -0.0316*T+5.1955*t 4.2

where T= calcination temperature (^{o}C)

t= calcination time (hour)

4.2.2 Statistical Analysis on Water Absorption

From the overview of the result obtained in Table 4.3, all values of water absorption are within the acceptable range of the factory i.e. 0.15-0.35 cm/min. The respective values of setting time and compressive strength at maximum water absorption, 0.34cm/min, are

respectively 16.8 minutes and 5.1987MPa. The setting time value is well above the lower limit but the compressive strength value is only a little above the lower allowable compressive strength for casting, 5MPa.Water absorption values greater than the upper limit implies higher diffusion rate of water through the mould due to high porosity. This will result in low strength of the mould hence short life span. Water absorption value lower than the lower limit implies low affinity of the mould to suck in water which will result in low production rate and poor quality product.

In order to see the effect of the independent variables (calcination temperature and calcination time) on the response (water absorption), graphs are presented below. As can be seen from the graphs, the effect of calcination time on water absorption is not significant while the calcination temperature affects the water absorption inversely.

The water absorption values were used to develop a mathematical model that correlates the water absorption to the calcination temperature and calcination time. Design Expert software version 7.0.0 was used for the regression analysis of the experimental data and also for evaluation of the statistical significance of the equation developed.

The model was tested for adequacy by analysis of variance. The regression model was found to be highly significant with the correlation coefficients of determination of R-Squared (R^2), adjusted R-Squared and predicted R-Squared having a value of 0.9726, 0.9696 and 0.9621, respectively.

Table 4.5: Analysis of Variance (ANOVA) for Water absorption

Source	Sum of square	Df	Mean square	F value	p-value prob> F	Remark
Model	0.021	2	0.010	319.87	<0.0001	Significant
A-temp	0.021	1	0.021	638.61	*<0,0001*	
B-time	3,643E-005	1	3.643E-005	1.13	*0.3009*	
Residual	5.781E-	18	3.212E-005			

	004					
Lack of fit	2.581E-004	6	4.301E-005	1.61	0.2263	Not significant
Pure error	3.200E-004	12	2.667E-005			
Cor Total	0.021	20				

The Model F-value of 319.87 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise.Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model.

The "Lack of Fit F-value" of 1.61 implies the Lack of Fit is not significant relative to the pure error. There is a 22.63% chance that a "Lack of Fit F-value" this large could occur due to noise.

Development of the Regerssion Model

The model equation that correlates the response (water absorption) to the calcination process variables, calcination temperature and time is:

Water absorption (cm/min.) =1.743- 0.0101*T-4.2677*t…….4.3

Where T=temperature (^{o}C)

t=time(hour)

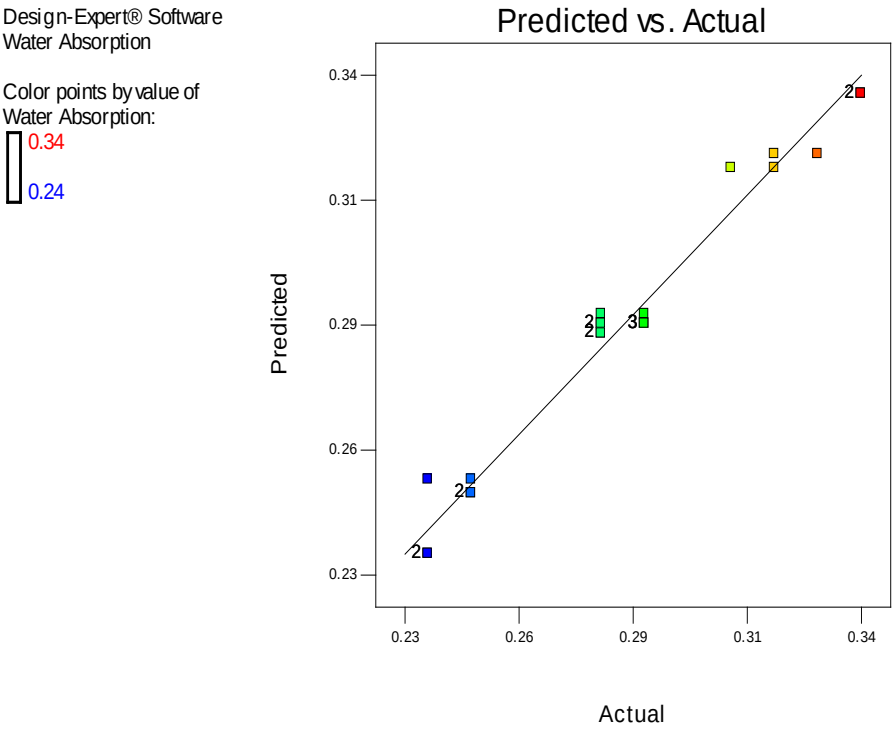

Fig.4. 6 Predicted versus Actual Setting time

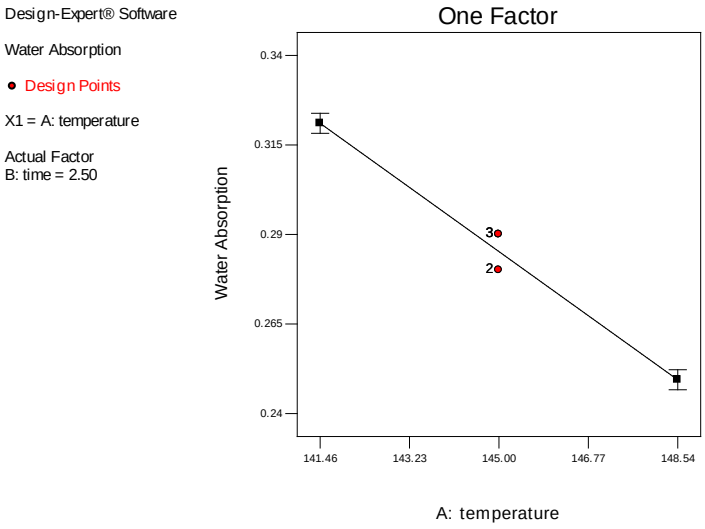

Fig.4. 7 Effect of Calcination temperature on Water Absorption

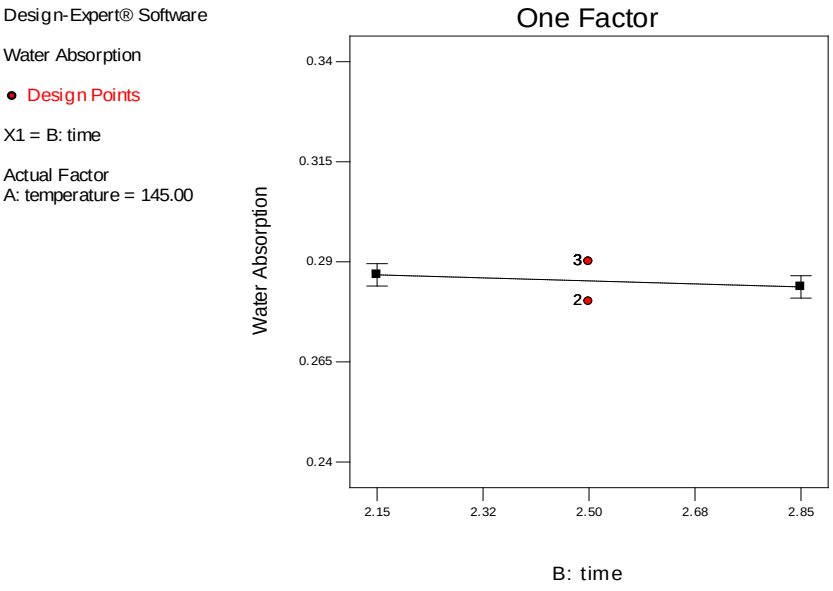

Fig.4. 8 Effect of Calcination time on Water absorption

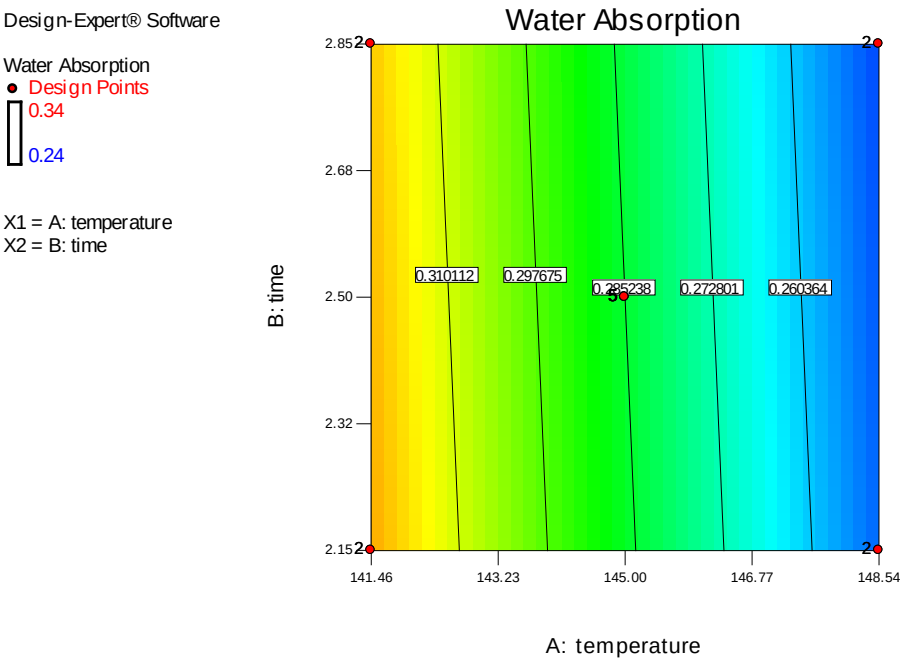

Fig.4. 9 Contour plots of effect of calcination temperature and calcination time on water absorption

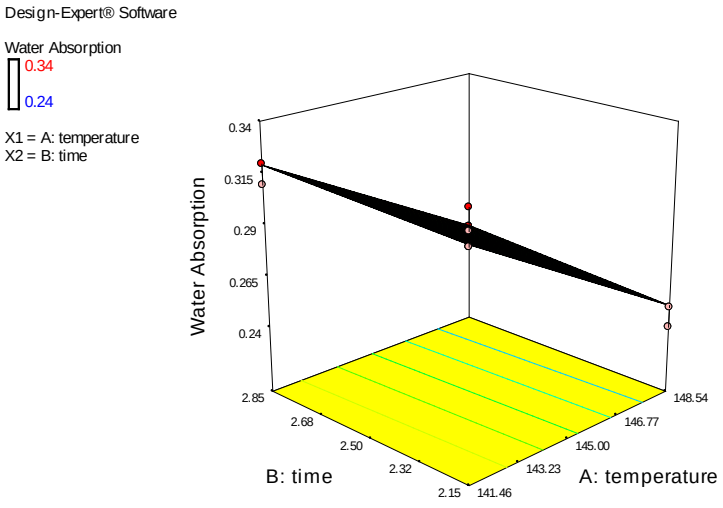

Fig.4. 10 Surface plots of effect of calcination temperature and calcination time on water absorption

4.2.3 Statistical analysis on compressive strength

The compressive strength is the most import response variable for recycling the discarded gypsum mould, it directly affects the mould life span upon casting. From the overview of the result obtained in the experiment,Table 4.3, the highest value of compressive strength

obtained is 5.7010 MPa at calcination temperature of 148.54^{o}C and calcination time of 2.15 hours. At this point the value of the setting time and water absorption is respectivly 14.6 minute and 0.25 cm/min.The water absorption value is well within the acceptable range of the factory i.e 0.15-0.35cm/min. But the setting time value 14.6minutes is below the lowet accepatble setting time for casting i.e 15 minute. Therfore recycling at this point is not acceptable.The lowest compressive strength obtained is 4.9886 MPa at calcination temperature 141.46 ^{o}C and calcinaton time of 2.15 hours.

Contour plots and 3-D response surfaces of the results obtained were constructed by plotting the response (compressive strength) on the Z-axis against the two independent variables (calcination temperature and calcination time), which is helpful to understand the main and the interaction effects of these two factors.

The model was tested for adequacy by analysis of variance. The regression model was found to be significant with the correlation coefficients of determination of R-Squared (R^2), adjusted R-Squared and predicted R-Squared having a value of 0.9513, 0.9427 and 0.9133, respectively.

Based on the analysis of variance, the setting time was not affected by interaction effects of the process variables. The factor that affect the compressive strength significantly is the calcination time. Figure 4.11 shows the effect of calcination time on compressive strength. It can be seen that with increasing calcination time the compressive strength increases significantly. The effect of calcination temperature on compressive strength is not significant and the result is shown in figure 4.13

Table 4.6: Analysis of Variance (ANOVA) for Compressive strength

Source	Sum of square	Df	Mean square	F value	p-value prob> F	Remark
Model	0.67	3	0.22	110.76	<0.0001	Significant
A-temperature	0.34	1	0.34	168.47	*<0.0001*	
B-time	0.031	1	0.031	15.25	*<0.0001*	
AB0.30	1	0.30	148.54	<0.0001		
Residual	0.034	17	2.012E-003			
Lack of fit	0.023	5	4.655E-003	5.11	0.009	significant
Pure error	0.011	12	9.102E-004			
Cor Total	0.70	20				

The Model F-value of 110.76 implies the model is significant. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. Values of "Prob > F" less than 0.0500 indicate model terms are significant. In this case A, B, AB are significant model terms. Values greater than 0.1000 indicate the model terms are not significant. If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve your model. The "Lack of Fit F-value" of 5.11 implies the Lack of Fit is significant. There is only a 0.96% chance that a "Lack of Fit F-value" this large could occur due to noise.

Development of the Regerssion Model

The model equation that correlates the response (Compressive strength) to the calcination process variables, calcination temperature and time is:

Compressive Strength = -56.8872+0.42769*T+22.54231*t-0.15461* T* t.....5.4

Where T=temperature (^{o}C)

t=time (hour)

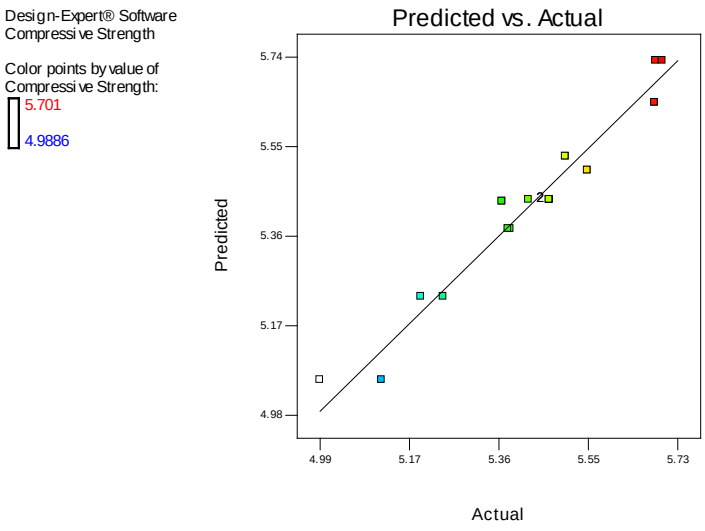

Fig.4. 11 Predicted versus Actual Compressive strength

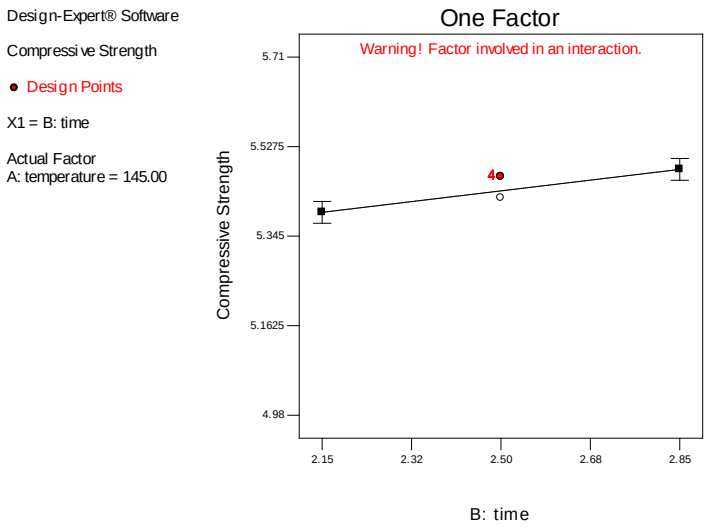

Fig.4. 12 Effect of Calcination time on Compressive Stregth

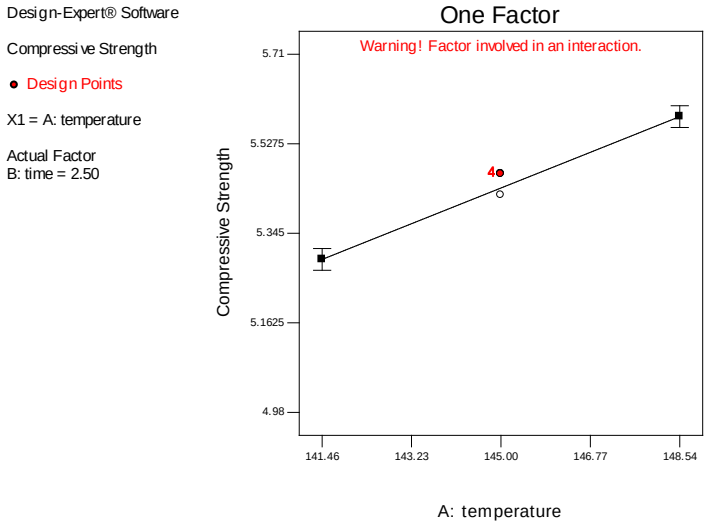

Fig.4. 13 Effect of Calcination temperature on Compressive Stregth

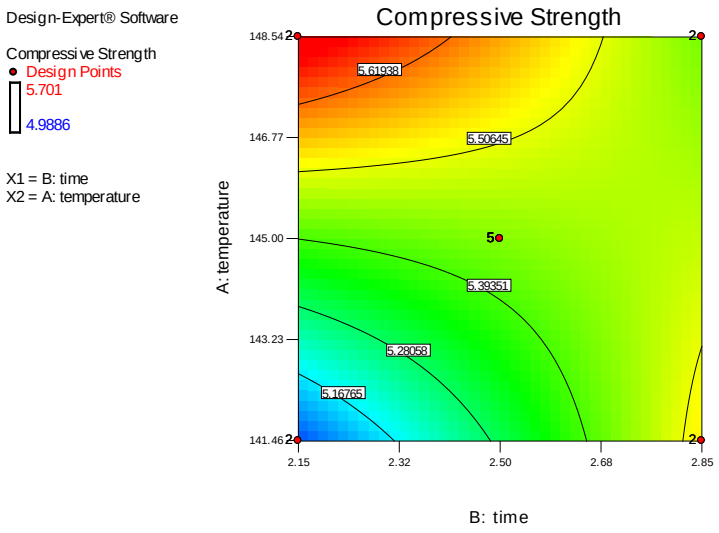

Fig.4. 14 Contour Plots of Effect of Calcination Temperature and Calcination Time on Compressive Strength

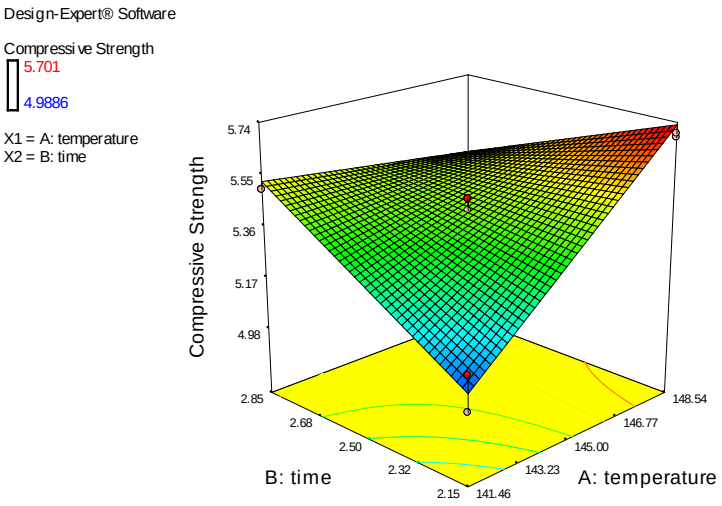

Fig.4. 15 Surface Plots of Effect of Calcination Temperature and Calcination Time on Compressive Strength

4.2.4 Optimization of Process Variables

In order to find the optimal working condition for recycling plaster of Paris from waste gypsum mould, the upper and lower limits of the independent and the response variables

along with the criteria for optimization are given below. Based on the criteria numerical and graphical optimization are made using expert design 7.0.0.

Table 4.7: Criteria for optimization

Variable	Unit	Lower limit	Upper limit	Goal
Temperature	^{o}C	140	150	In range
Time	Hour	2	3	In range
Setting time	minutes	15	19.3	In range
Water absorption	cm/min	0.15	0.35	In range
Compressive strength	MPa	5.00	5.7010	Maximize

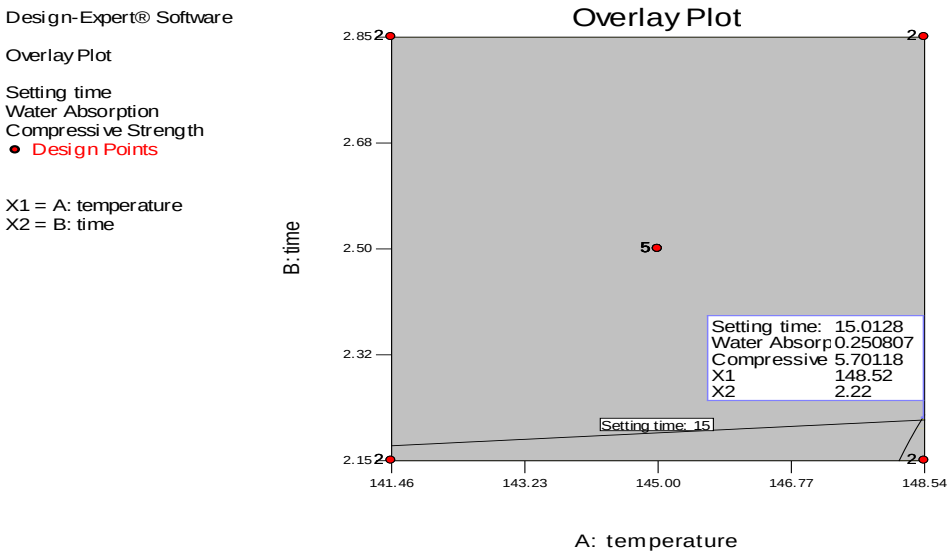

Fig.4.16 Optimization of Process Variables

Discussion

The optimization analysis shows that an optimized result of setting time 15 minutes, water absorption 0.25 cm/minutes and compressive strength value of 5.7010MPa is obtained at a calcination temperature of 148.52^{o}C and calcination time of 2.22 hour.

4.3 Blending Analysis

The virgin and the recycled plaster of pairs are mixed with different proportions and the response variables are measured for each sample. The recycled plaster of Paris used for blending analysis is obtained from the optimization analysis (calcination temperature of 148.52^{o}C, calcination time of 2.22 hours and with setting time of 15minutes, water absorption of 0.25 cm/min and compressive strength of 5.7010 MPa). Blending results are given below in Table 4.8.

Table 4.8: Results of Blending Test

No.	% Recycled	setting time(min)	Water absorption (cm/min)	Compressive strength, (MPa)
1	100	16	0.23	6.1025
2	75	17	0.24	7.6458
3	60	19	0.28	8.6956
4	50	20	0.3	6.5020
5	40	21	0.27	6.3421
6	0 (100% virgin)	15	0.25	5.7010

Discussion

As can be seen from the experimental result in Table 4.8, blending generally increases the setting time upon casting. The maximum setting time achieved in the experiment for a blend

is 21 minutes when 60% virgin plaster is mixed with 40% recycled plaster. At this point the corresponding values of the water absorption and compressive strength are respectively 0.27 cm/min and 6.3421 MPa. A setting time value greater than 20 minutes is not recommended for casting based on the incoming inspection standard of the factory. The minimum setting time is 15 minutes when 100% virgin plaster is used. For all blending proportions, the setting time is well within the range of the standard developed by the factory, except at 40% recycle and 60% virgin blend, and the selection of the optimal working condition depends on the other response variables, the water absorption and the compressive strength.

The water absorption increases as the proportion of the recycled plaster increase until a recycled proportion of 50% and then it decrease. The maximum water absorption of the blending experiment is 0.3 Cm/min for 50% recycled plaster and 50% virgin plaster. The water absorption is highest for 100% recycled plaster, 0.315 cm/min. All the values of the water absorption are within the acceptable range developed by the factory for incoming inspection.

The compressive strength of the blending experiment shows that the compressive strength of the blend is generally higher than that of the virgin plaster of Paris. As the proportion of the virgin plaster of Paris increases from 0 to 60%, the compressive strength increases from 6.1025 to 8.6956MPa and then it decreases to 5.7010 MPa for a 100% virgin plaster of pairs. At 60% recycled proportion, the compressive strength is 8.6956 MPa and the setting time and the water absorption are respectively 19 minutes and 0.28Cm/min. A higher value of compressive strength means a longer life span of the plaster mould for slip casting. Therefore the number of casts with this blend will be greater than with a mould made totally from virgin plaster. If needed to blend the recycled and the virgin plaster of Paris, it is at this point that we should operate taking into consideration the economic feasibility of the work.

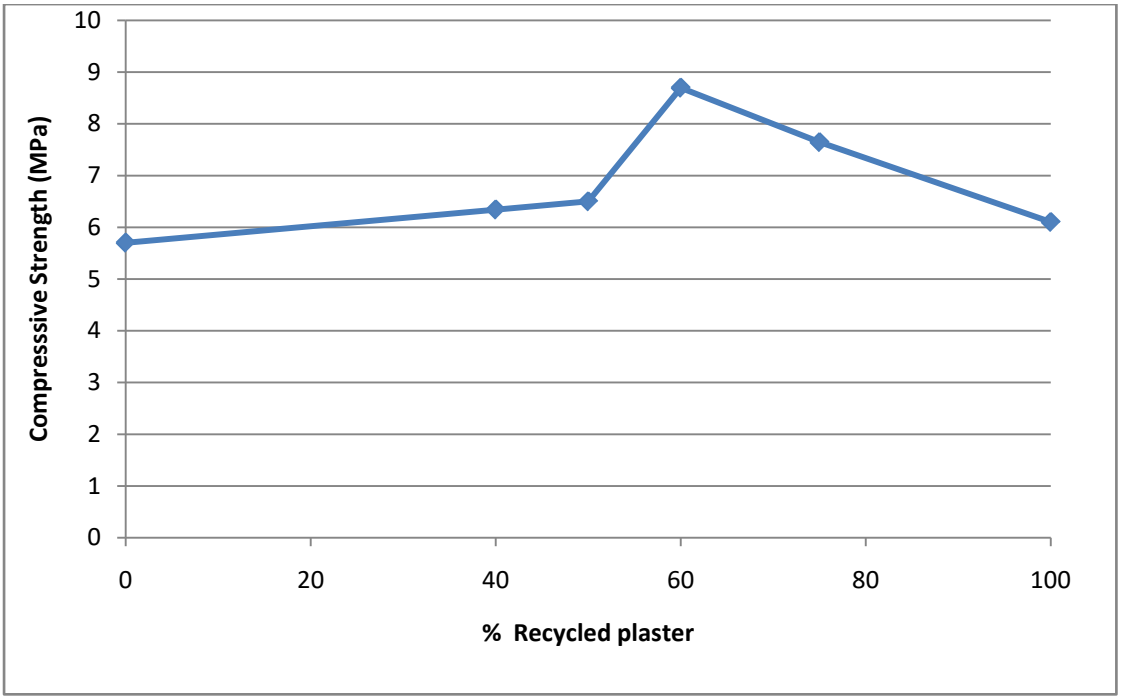

Fig.4. 17 Effect of blending on Compressive Strength

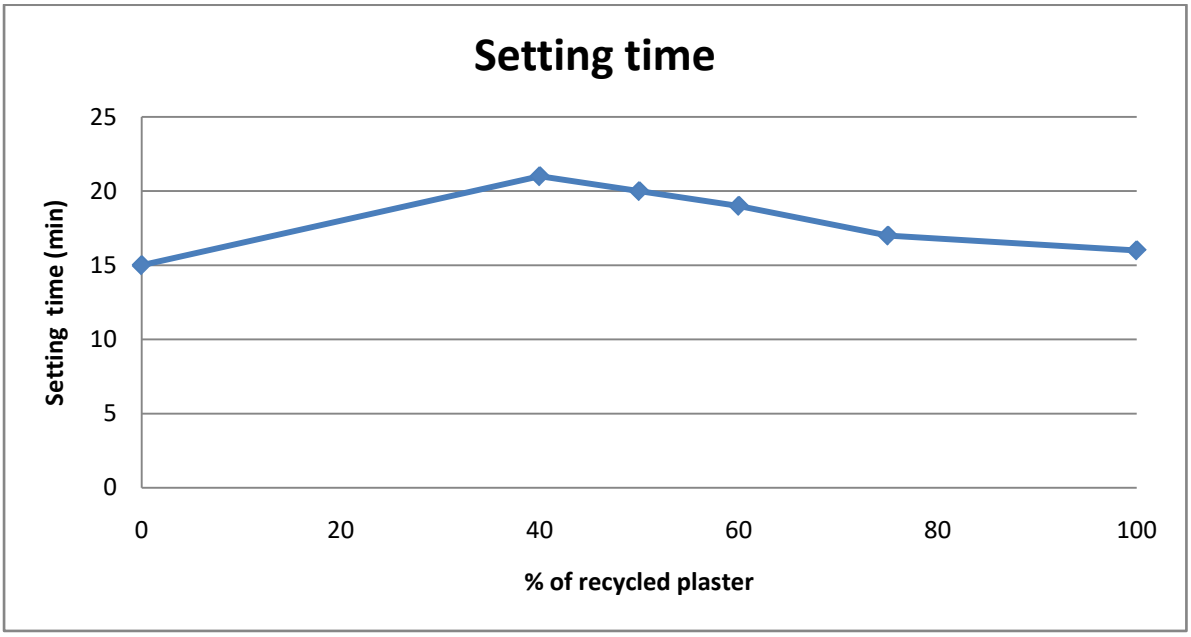

Fig.4. 18 Effect of blending on Setting Time

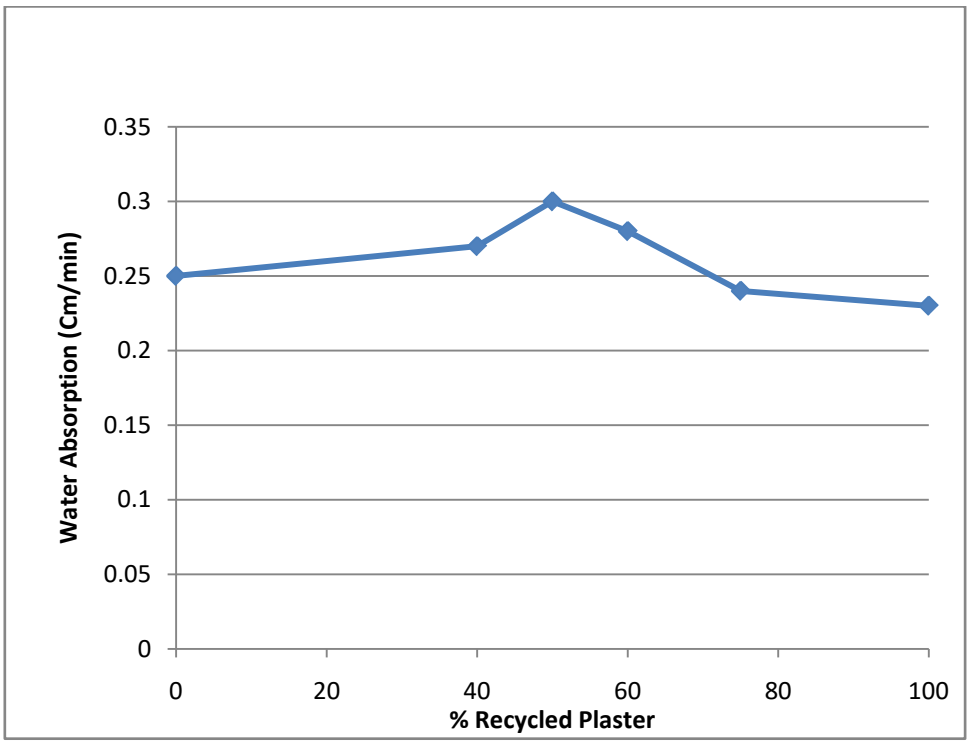

Fig.4. 19 Effect of blending on Water Absorption

5. DESIGN OF PROCESS AND EQUIPMENTS

POP is produced by calcining gypsum as per the equation below:

$$CaSO_4.2H_2O \longrightarrow \beta\ CaSO_4.\ \frac{1}{2}\ H_2O + \frac{3}{2}\ H_2O \quad \ldots\ldots\ldots\ldots\ldots\ldots\ldots 5.1$$

ΔH= + 597,200 kJ/T POP

ΔH= +142,600 kcal /T POP

ΔH= + 165.9 kWh/T

An overall material balance is derived using the stoichiometric principle combined with data concerning impurities in raw material collected from units.

Production of POP is an endothermic reaction as indicated in the equation above. The thermal energy requirement and balancing should consider physical moisture in gypsum, combustion efficiency, heat transfer efficiency from combustion chamber to calciner material, insulation and other factors. [2] Considering the many variable factors that influence energy consumption, energy balance is done as follows.

5.1. Overall Material Balance For producing 1ton of POP

Material Balance Basis:

$CaSO_4.2H_20$+Impurities (As a basis) = 1 ton

Assumptions:

- Assume Moisture Content = 0.5%
- Impurities other than gypsum = 8.4%
- Grinding and other losses=3.6%

Material Balance:

H_2O loss (physical) = 0.005*1=0.005ton

H_2O loss (Chemical) = $\frac{molecular\ \ wt.of\ gypsum\ -molecular\ \ weig\,ht\ of\ POP}{molecular\ \ weig\,ht\ of\ gypsum}$*1 ton……....5.2

$=\frac{172.17-145.15}{175.15}$*1ton

=0.157ton

Product Grinding losses including removal of impurities = 0.12*1 ton= 0.12 ton……5.3

Specific Product obtained = 1ton-0.157ton-0.12 ton

= 0.718 ton POP / ton Gypsum……...…..…….5.4

The overall material balance indicates the major difference between raw material utilized and product obtained is due to removal of physical and chemically bound moisture followed by partial removal of impurities.

5.2. Energy Balance

In POP manufacturing thermal energy is used in calcinations and electric energy is used in driving motors, conveyors etc. Electrical energy constitutes only 5% of the total energy consumption and thermal energy the rest 95 %.

Considering the calcination process to take place in the Rotary Drum calciner which is horizontal drums, made up of mild steel, rotating along the horizontal shaft at slow speed of around 10-12 rpm. The batch capacity of drum is in the tune of 1 ton and the batch time varies from 1.5 hrs to 4 hrs, depending on desired quality & end use of product. For this specific purpose the calcination time is 2.22 hours and thermal energy is supplied from electricity. Rotary drum calciner is cheap and commonly used for low production capacity processes, the total thermal efficiency is as low as about 55%. [2]

From the stoichiometry, we have;

ΔH= + 597,200 kJ/ton POP

$E_{thermal} = \frac{\Delta H}{0.55} = \frac{+597{,}200\,KJ/T}{0.55}$=1,085,818 kJ/ton POP……...…..……5.5

$E_{thermal} = \frac{1085818\,KJ/T}{0.553600}$ = 301.6 kWh/ton POP…….....….5.6

$$E_{electrical} = \frac{5*301.6}{95} = 15.87 \text{ kWh/ton POP} \quad \ldots\ldots 5.7$$

The factory is using plaster of Paris in the sanitary ware line for casting of moulds and table wares line for forming process. When the factory produces in its full design capacity, the consumption of plaster of Paris is about 10 tons per week. The annual consumption of plaster of Paris is 430 ton assuming the factory works for 43 weeks per annum.

5.3. Process and equipment selection for the recycling of the discarded gypsum mould

The annual plaster of Paris consumption of the factory recently is around 430 ton for 300 working days per annum. Discarded plaster of moulds has been accumulated in open air dumping site since the establishment of the factory. Considering this fact and the expansion program of the factory to increase its production capacity, I propose a recycling plant of 750 tons per annum.

5.3.1. Materials and inputs

The main material inputs are discarded gypsum mould and packing materials. The packing material like the discarded mould can be reused for the recycling process

Table 5. 1: Estimated annual cost of material inputs

No	Description	Qty	Cost
1	Discarded mould	750 ton	-
2	packing material	750,000 pcs	-
	Total		-

5.3.2. Utilities

The major utilities required by the plant are electricity and water. Electricity is used both to drive machinery parts and as a source of thermal energy for calcination. The estimated annual requirement at 100% capacity utilization rate and the estimated costs are given in table below.

Table 5. 2: Annual utility requirement and estimated cost

Sr. No	Description	Qty (Annual consumption)	Cost per unit (Birr)	Total cost (Birr)
1	Electric power, kWh	11,902.5=(15.87*750)	0.58	6,903.45
2	Electric power (calcination), KWh	226,200=(301.6*750)	0.58	131,196
3	Water, m^3	7500=(10*750)	5.6	42,000
	Total			180,099.45

5.3.3. Process machinery and equipment

a. Process description

First the discarded moulds are cleaned of clay, sand, dust and deflocculated crystals deposited on the mould surface, the moulds are then manually crushed and the fine lumps are washed with water in the ball mill where the balls are removed. Half the volume of the mill is filled

with gypsum lumps and water about 40% the volume of the mill is added. The mixture is rotated at low rpm for about 5-10 minutes to remove the soluble deflocculates. The material is then dried for further processing. Drying takes place in rotary drum calciner at a temperature of 50^{o}C for 24 hours.

The dried gypsum is crushed in a ball mill where the particle size is reduced to less than 25mm.The cleaned, dried and crushed gypsum is stored in a storage silo before calcination. The calcination process takes place at a temperature of 148.55^{o}C in a rotary drum calciner for 2.22 hours. During the process of calcinations, the gypsum loses 75% of its moisture thereby making it hydroscopic The calcined POP after cooling is manually fed in the pulverize and grounded further to a particle size of 90-95% passing through 150µm. Aging of the mixture in the second storage silo then takes place for about 3-4 weeks before packing.

The technology of calcinated gypsum production is simple and some of the equipments can be locally manufactured and the rest acquired from India, China or Italy through commercial attaches.

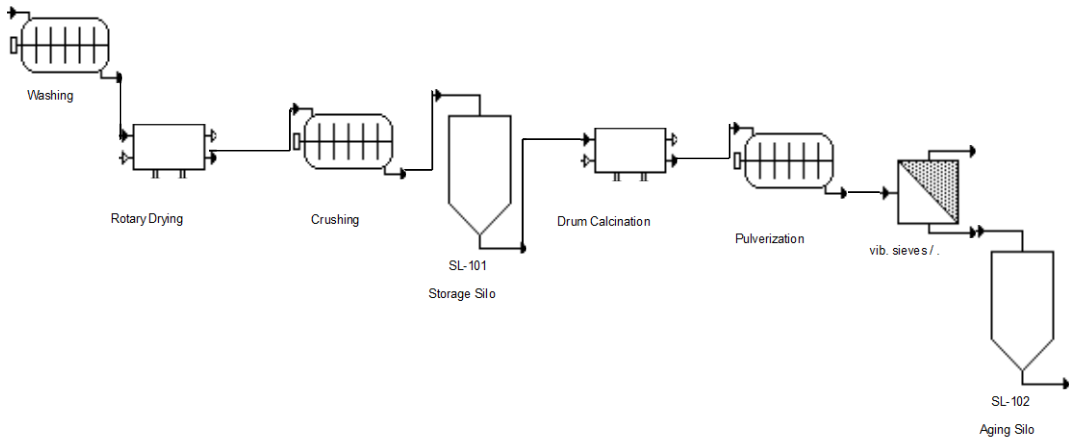

Fig.5. 1 Equipment layout for recycling plaster of Paris

b. Machinery and Equipment

The production equipment required by the plant and their estimated costs are given in Table 5.3.

Table 5. 3: Machinery and equipment requirement

Sr. No	Description	capacity	Unit	Qty	Total Cost (birr)
Sr. No	Description	No	Salary (birr)		
1	Rotary drum Calciner	10 ton	Required	Monthly	Annual
2	Crusher and Pulverizer machine	10 ton	900	1	10,800
3	Storage silo, locally manufactured	10 ton	pcs	2	24,000
2	Calcination operator	2	1,500		18,000
3	Packing	2	900		10,800
Total	Total	6	6,600		79,200
Employee's benefit (25% basic salary)		-	-		19,800
					72,000
2	Manual size reduction and cleaning	2	pcs	1	469,000
4	Vibrating sieves	-	Set	1	131,000
Total					696,000

Costs obtained from the internet (http://www.matche.com/EquipCost/Index.htm, retrieved on April 5, 2014)

C. manpower requirement

The total manpower requirement of the plant is 6 persons. Details of manpower and estimated annual labor cost are given in the Table 5.4 below.

Table 5. 4: Manpower requirement

Grand total	-	-	99,000

6. FINANCIAL ANALYSIS OF PLASTER RECYCLING PLANT

Total Capital Investment Cost Estimation

Typical factors for estimation of fixed capital cost for most chemical plant processing are considered for the estimation of capital investment. (Periasamy,1996)

	Fixed Capital Cost	
1	Purchased Equipment (delivered)(PE)	**696,000.00**
2	Purchased equipment installation, 39% PE	271,440.00
3	Instrumentation and controls, 12% PE	83,520.00
4	Electrical (installed), 10% PE	69,600.00
	Building, service and auxiliary, 10%	69,000.00
	Total direct cost (DC) =	**1,188,960.00**
	Engineering and supervision, 5% DC	59448.00
	Contingency, 8% DC	95116.8
	Total Indirect Plant Cost=	**154,564.8**

Fixed Capital Investment, FCI = Direct cost + Indirect cost

= 1188960.00+154564.8

FCI= 1,343524.8

Working Capital

Working Capital, 15% of FCI

Working capital = 0.15 x 1,343524.8 Birr

WC =201528.72

Total Capital Investment = Fixed Capital + Working Capital

TCI = 1,343524.8 +201,528.72

= **1,545,053.52 Birr**

Estimation of Total Production Cost (TPC)

Manufacturing Cost

A. Direct Production Cost

- Raw material = 0
- Operating labor = 99,000
- Utilities

- Electricity, (including 15% tax)

  = 138,100.00 Birr/year

- Water, (including 15% tax)

  = 42,000.00 Birr/year

- Maintenance and repairs, 4% FCI

  = 0.04 x 1,343524.8

  = 53,741.00 Birr/year

- Laboratories, 10% operating labor

  = 0.10 x 99,000

  =9,900.00 Birr/year

Direct production cost = RM cost + operating labor + utilities + maintenance & repairs +laboratory

= 0+ 99,000.00 +138,100.00+42,000.00+53,741.00 + 9,900.00

DPC =342,741.00 Birr

B. Fixed Charges

i. Depreciation, 10 % FCI = 134,352.48 Birr

ii. Capital charge (local tax), 1% FCI = 13435.24 Birr

iii. Insurance, 0.4% FCI = 5374.09 Birr

Fixed charge = 153161.81 Birr,

C. Plant Overhead, 10% TPC

Total Manufacturing cost = A+ B +C

= 269,739.09+ 153,161.81 + 0.1*TPC

=422,900.90+ 0.1 TPC

General Expenses

a. Distribution and selling cost, not required

b. Research and development cost, not required

Total general expense = 0

Total production cost = Manufacturing cost + general expenses

TPC = 422,900.90 + 0.1 TP`C

0.9 TPC =422,900.09

TPC = 469,889.88 Birr/year

Estimation of Total Income from the plaster of Paris regeneration Plant

According to the design, a plaster regeneration plant of 750 ton per annum is established in the compound of the factor. Waste gypsum mould discarded at the end of the process since the establishment of the factory is used as input for the process. Taking a total of 30% loss in the whole process, the net production of plaster of Paris is 525 ton per year.

Income generated from the regenerated plaster of Paris at the current local price of plaster of Paris i.e. 3 Birr/kg is:

3.0 Birr/kg* 525ton/Yr*1000 kg/ton=1,575,000.00

Economic Evaluation

1. Gross and Net Earning

Total income = Total annual sale

Gross Annual Earning = Total Income – Total production cost

= 1,575,000 – 469,889.88

1,105,110.2= **Birr/year**

Net Annual Earning = Gross Earning – Tax

N.B Assume income tax to be 35%

Net Annual Earning = 1,105,110.2 – (0.35 ×1,105,110.2)

= **718,321.7**

2. Return on Investment (ROI)

Cash Flow

The cash flow for each year is listed below assuming the plant to operate at 85% capacity at first year, 90% at the 2nd year, 95% at the 3rd year and 100% for the next 7 years

Table 6. 1: Cash flow rates for ten years

Year	Cash flow, Birr
1st	610,573.44
2nd	646,489.53
3rd	682,405.61
4th up to 10th	7*(718,321.7)= 5,028,251.9

Cumulative cash flow for ten years (Total profit) = 6,967,720.40 Birr

The average net profit per year = 696,772.04Birr

Total Capital Investment (TCI) = Fixed capital +working capital = 1,545,053.52 Birr

If the plant depreciates over 10 years

Depreciation per year = 1,545, 053.52/10 = 154,505.35birr

$$\text{ROI} = \frac{(\text{Total profit} - \text{depreciation}) \times 100}{\text{Total capital} \times \text{year}}$$

$$= \frac{(6{,}967{,}720.4 - 154{,}505.35) \times 100}{1{,}545{,}053.52 \times 10}$$

ROI = 44.09 %

3. Pay Back Period (PBP)

$$\text{PBP} = \frac{\text{Total fixed capital}}{\text{Average annual profit}}$$

$$= \frac{1{,}343{,}524.8}{696{,}772.04}$$

PBP = 1.92 years

7. CONCLUSION AND RECOMMENDATION

7.1. Conclusion

In this research, recycling of plaster of Paris from discarded gypsum moulds has been investigated. Two calcinations parameters affecting the property of the plaster of Paris; calcinations temperature and calcinations time have been studied. The outputs of the experiment conducted have been analyzed by measuring the setting time, the compressive strength, the water absorption and other physical properties of the plaster of Paris.

The result obtained shows that there is a great potential of recycling plaster of Paris from discarded gypsum moulds and using it for casting process in ceramic production mainly because it decreases the production cost of buying virgin plaster of Paris and also results in the development of environmentally friendly process by decreasing the waste gypsum moulds that go to open air dumping.

Based on the experimental result obtained, all values of water absorption are within the acceptable range of the factory while setting time values below 15 minutes are not acceptable for casting process. Based on the incoming inspection criteria of the factory, compressive strength value below 5MPa is not acceptable for casting. Optimum values of 15 minute setting time, 0.25cm/min water absorption and 5.7010MPa compressive strength are obtained at a calcinations temperature of 148.5^{o}c and calcinations time of 2.22 hours.

The mole of water of crystallization of the sample before calcination was found to be 1.943 and this proved that it was in a dihydrate state since mole of water of crystallization of dihydrate gypsum is 2. Following the same procedure, calcination of the sample at a

temperature of 148.5^{o}C for 2.2 hours resulted in mole water of crystallization of 0.45 and this proves conversion to hemihydrate state since mole water of crystallization of hemihydrate plaster is 0.5.

The blending experiment showed that the best result is obtained when 60% recycled plaster of pairs is mixed with 40% virgin plaster of pairs. At this point, the compressive strength is 8.6956 MPa and the setting time and the water absorption values are respectively 19 minutes and 0.28cm/min. A higher value of compressive strength indicates a longer span of the plaster mould for slip casting.

Using the optimum condition determined experimentally and the existing condition of the plant, plaster of Paris recycling plant has been proposed and selection of the equipments to be used in the plant is also done.

Finally the proposed recycling plant is financially analyzed and it is found that it is with a pay pack period of two years which makes the implementation of the plant feasible.

7.2. Recommendation

The cost of production in ceramics manufacturing can be minimized by reducing the cost of production inputs and the cost incurred for waste disposal. The ever increasing cost for virgin plaster of pairs which is mostly imported from abroad is one of the factors that severely affect the profitability of Tabor ceramic products manufacturing Share Company.

Depending on the results obtained in this research, available resources and technical feasibility, the following recommendations are suggested:

- In order to obtain realistic results, experiments should be conducted at industrial scale at similar conditions as those prevailing in the actual process conditions. While the parameters of the recycled plaster of Paris is within the acceptable range of the factory at the incoming inspection stage, actual casting of ceramic products should be conducted using the moulds made from recycled plaster of Paris.
- The compressive strength is the most important variable for recycling the discarded gypsum mould. It directly affects the mould life span upon casting.Further study should be conducted to see the possibility of enhancing the performance of the recycled plaster of Paris by adding additives to increase the compressive strength of the moulds and the properties of the plaster of Paris when recycled more than once.
- The calcinations temperature and calcinations time for best result in recycling plaster of pairs from waste gypsum moulds may depend on both the type of virgin plaster of pairs

used and the type of casting used in the process. Therefore the results found in this work may not be best applicable for other areas.

- In addition to recycling and using the discarded gypsum moulds for ceramic casting, further studies should be conducted to see the potential to use it in areas like cement industry and conditioning of acidic soil.

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9. APPENDICES

A 1: Laboratory Analysis Results

Factors	Run#1		Run#2	
Calcination Temperature	145	^{o}C	141.6	^{o}C
Calcination Time	2.5	Hour	2.85	Hour
	Run#3		Run#4	
Calcination Temperature	141.46	^{o}C	148.54	^{o}C
Calcination Time	2.15	Hour	2.15	Hour
	Run#5		Run#6	
Calcination Temperature	145	^{o}C	145	^{o}C
Calcination Time	2.50	Hour	2.00	Hour
	Run#7		Run#8	
Calcination Temperature	150	^{o}C	145	^{o}C

Calcination Time	2.00	Hour	2.00	Hour
	Run#9		Run#10	
Calcination Temperature	145	^{o}C	140	^{o}C
Calcination Time	2.50	Hour	2.50	Hour

Experimental Design Run Sheet

Factors	Run#11		Run#12	
Calcination Temperature	145	^{o}C	14	^{o}C
Calcination Time	3.0	Hour	2.50	Hour
	Run#13		Run#14	
Calcination Temperature	150	^{o}C	141.46	^{o}C
Calcination Time	2.50	Hour	2.85	Hour
	Run#15		Run#16	
Calcination Temperature	141.46	^{o}C	148.54	^{o}C
Calcination Time	2.15	Hour	2.85	Hour
	Run#17		Run#18	
Calcination Temperature	148.54	^{o}C	145	^{o}C
Calcination Time	2.85	Hour	3.00	Hour
	Run#19		Run#20	
Calcination Temperature	148.54	^{o}C	145	^{o}C
Calcination Time	2.15	Hour	2.50	Hour
	Run#21			
Calcination Temperature	140	^{o}C		
Calcination Time	2.50	Hour		

No	Specimen dimension (cm^2)	Type	Force (kN)	Pressure (MPa)
1	10.5*2.5=26.25	75 virgin	22.8	8.6857
2	8.5*2.3=19.55	40% Virgin (water immersed)	17	8.6956
3	7.5*2.6=19.50	100 recycled	11.9	6.1025
4	2.8*8.9=24.92	25% virgin (water immersed)	13.9	5.6458
5	9.2*2.6=23.92	40% recycled	14.7	6.1454
6	8.6*2.7=23.22	100% virgin	15.6	6.7183
7	9*2.9=26.1	25% virgin	12	4.5977
8	9*2.7=24.3	50% virgin	15.8	6.5020
9	10.5*2.8=29.4	100% virgin (water immersed)	20.8	7.0748

B. 1: Blending and compressive strength test

B. 2: Blending and setting time

No	% recycled in the mix	setting time(min)
1	100	16
2	50	20
3	75	17
4	40	21
5	0 (100% virgin)	22

B. 3: Blending and water absorption test

No	% recycled in the mix	Water absorption for 6 cm bar (min.)	Water absorption(velocity) (cm/min)
1	100	16	0.315
2	50	20	0.3
3	75	25	0.24
4	40	27	0.27
5	0 (100% virgin)	23	0.26

C. Determination of mole of water of crystallization

Mass of Dish, Md

Mass of Dish + mass of powder, Mdp

After heating for six hours at 180^0C

Mass of Dish + Dried Powder, Mdd

Mass of water lost, Mw=Mdp-Mdd

Molar mass of water = 18 g/mol

Mole of water lost, Mow = Mdp-Mdd /18

Mass of anhydrous CaSO4=Mad = Mdd – Md

Molar mass of CaSO4 = 136.15 g/mol

Mole of anhydrous $CaSO_4$ = Mad /136.15

To determine x in $CaSO_4 .xH_2O$ (i.e., ratio of mole of water to $CaSO_4$):

x = Mole of water lost/ Mole of anhydrous $CaSO_4$

D. 1: Physical and chemical properties of plaster of Paris (calcium sulphate, hemidydrte)

Molecular formula	$CaSO_4.1/2H_2o$	Appearance	White powder
Molecular mass	145.15	Odor	No oder
Specific gravity	2.32g/mL@20 ^oc	Odor Threshold	N/A
Vapour density(air=1)	N/A	Solubility	Slightly soluble
Melting point	N/A	Evaporation rate	N/A
Boiling point	N/A	Partition coeff.	N/A
Vapour pressure(20 ^oc)	N/A	Ph	N/A
Flash point	N/A	LEL	N/A
Autoiginitin temp.	N/AA	UEL	N/A

D. 2: Physical properties of Gypsum and POP

Property	Gypsum	POP
Molecular weight	172.17	145.15
Density	2.31	2.62-2.64
Hardness,Mohs	1.5	
Water of crystallization	20.92	6.21
Water solubility@20^{o}c g/100g solution	0.21	0.88

E. 1

E. 2: Common Plaster Additives

Additive	Function
Starch	Used in plaster board to protect the physical bond between the gypsum crystals and the cardboard during drying.
Ground gypsum	Provides many sites at which gypsum crystals can grow, thus accelerating the setting rate.
Lignosulphonates	Improves the flow of the slurry so less water is required, resulting in a denser plaster.
Potasium sulphate	Causes the gypsum to precipitate out quicker due to a common ion effect.
Detergent	Forms foam in the mix, resulting in a less dense plaster. The detergent chosen must form foam in hard water (i.e. water containing a high concentration of calcium ions).

F. 1: Ball mill for crushing and pulverization

Model	Φ1500×5700
Diameter of cylinder (mm)	1500
Length of cylinder (mm)	5700
Speed of bucket (r/min)	27
Max. input size (mm)	25
Output size (mm)	0.074-0.4
Capacity (t/h)	5-10
Motor power (kw)	130
Weight of ball (t)	12
Weight (T)	25
Plant Image	

Source: Shanghai Zenith Mining and Construction Machinery Co., Ltd., 2013

DECLARATION

I declare that the thesis for the M.Sc. degree at the University of Addis Ababa, hereby submitted by me, is my original work and has not previously been submitted for degree at this or any other university, and that all resources of materials used for this thesis have been duly acknowledged.

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Date of Submission:

This thesis has been submitted for examination with my approval as a university advisor.

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Signature:

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