

Addis Ababa University

School of Graduate Studies

Addis Ababa Institute of Technology
School of Chemical and Bio Engineering

Optimization of Gypsum Processing Parameters For the Production of Improved Quality Chalk

By

Gemechu Bushu

A Thesis Submitted to the School of Graduate Studies of Addis Ababa University, Institute of Technology, in Partial Fulfillment of the Requirements for the Degree of Masters of Science in Chemical Engineering (Process Engineering)

Advisor

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Approved by Board of Examiners

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TABLE OF CONTENTS

LIST OF ABBREVIATIONS v

LIST OF TABLES vi

LIST OF FIGURES vii

ABSTRACT viii

1 INTRODUCTION 1

1.1 Background . . . 1
1.2 Statement of the Problem . . . 1
1.3 Objective . . . 2
1.3.1 General Objective . . . 2
1.3.2 Specific Objectives . . . 3
1.4 Significance of the Study . . . 3

2 LITERATURE REVIEW 4

2.1 The Structure of Gypsum System . . . 4
2.2 Geological Occurrence and Abundance of Gypsum . . . 4
2.3 Occurrence of Gypsum in Ethiopia . . . 5
2.4 Production of POP and Chalk in Ethiopia . . . 6
2.4.1 Production of Plaster of Paris . . . 6
2.4.2 Production of Chalk . . . 7
2.5 The Physico-Chemical properties of Gypsum . . . 8
2.6 Technology Selection and Production Process of POP . . . 10
2.6.1 Size Reduction Process . . . 10
2.6.2 Kettle Calcinating . . . 10
2.6.3 Plaster of Paris (POP) . . . 11
2.7 Gypsum Dehydration Sequence . . . 13
2.8 Technology Selection and Production Process of Chalk . . . 15
2.9 Different Processing Parameters on Chalk . . . 18
2.10 Other Possible Raw Materials to Produce Chalk . . . 19
2.11 Application of Gypsum . . . 19
2.12 Environmental Implications . . . 20

3 MATERIALS AND METHODS 21

3.1 Time and Place of the Study . . . 21
3.2 Materials . . . 21
3.3 Methods . . . 21
3.3.1 Source and Preparation of Raw Materials Sample . . . 21
3.3.2 Size Reduction of the Sample . . . 22

3.3.3 Calcinations Procedure . . . 22
3.3.4 Mixing Procedure . . . 22
3.3.5 Moulding Preparation . . . 23
3.3.6 Drying Procedure . . . 23
3.4 Proximate Determination . . . 23
3.4.1 Determination of Moisture Content . . . 23
3.4.2 Determination of Bulk Density . . . 24
3.4.3 Determination of Breaking Strength . . . 24
3.5 Experimental Design of Process Parameters . . . 24
3.6 Factors Affecting the Quality of the Product . . . 26
3.6.1 Experimental Set-up . . . 26

4 RESULTS AND DISCUSSION **28**
4.1 Characterization of the Raw Materials . . . 28
4.1.1 Physical Characterization . . . 28
4.1.2 Chemical Characterization . . . 28
4.2 Statistical Results . . . 29
4.2.1 Optimization using Desirability . . . 30
4.2.2 Development of Regression Model Equation . . . 32
4.3 Data Analysis of Process Parameters . . . 33
4.3.1 Data Analysis for Bulk Density . . . 34
4.3.2 Data Analysis for Breaking Strength . . . 35
4.3.3 Diagnostic test . . . 36
4.3.4 Effect of Processes Parameters on the Responses . . . 37
4.3.5 Optimization of the Process Parameters . . . 42
4.3.6 Validation of Optimum Results . . . 43

5 FEASIBILITY STUDY OF IMPROVED QUALITY CHALK **45**
5.1 Product Description . . . 45
5.2 Quality Control . . . 46
5.3 Material and Energy Balance . . . 47
5.3.1 Material Balance . . . 47
5.3.2 Energy Balance . . . 51
5.4 Design of Equipment . . . 53
5.5 Financial Analysis . . . 54
5.5.1 Total Capital Investment . . . 54
5.5.2 Total Product Cost . . . 55
5.5.3 Profitability Measurements . . . 57
5.6 Plant Location . . . 58

6 CONCLUSION AND RECOMMENDATION **59**
6.1 Conclusion 59
6.2 Recommendation 60

REFERENCES **61**

APPENDICES **65**

Appendix A: Experimental Design Run Sheet **65**

Appendix B: Statistical Results Tables **65**

Appendix C: Statistical Results Figures **65**

LIST OF ABBREVIATIONS

2FI	Two Factor Interaction
3-D	Three-dimensional
ANOVA	Analysis of Variance
AI	Anhydrite I
AII	Anhydrite II
AIII	Anhydrite III
CCD	Central Composite Design
CI	Confidence Interval
Cp	Specific Heat Capacity
CT	Calcinations Temperature
C.V	Coefficient of Variation
D	Dehydrate
Df	Degree of Freedom
EMDPE	Educational Material Production and Distribution Enterprise
FCCD	Face Centered Composite Design
FGD	Flue Gas Desulphurization
GSE	Geological Surveys of Ethiopia
LSD	Least Significant Difference
MC	Moister Content
MS	Mean Square
POP	Plaster of Paris
PS	Particle Size
RSM	Response Surface Methodology
SS	Sum of Squares
Std. Dev.	Standard Deviation

LIST OF TABLES

1 Compositions Yogef gypsum occurrence[15] . . . 6
2 Production of POP in Ethiopia in consecutive ten years . . . 7
3 Imports of Plaster of Paris . . . 7
4 Production of Chalk in Ethiopia in consecutive five years . . . 7
5 Quantity of Chalk Imported . . . 8
6 Physical and chemical properties of gypsum . . . 9
7 Comparison of alpha and beta plaster . . . 12
8 Phases of gypsums . . . 13
9 Physical properties of gypsum, hemihydrate and anhydrite . . . 15
10 Hemihydrates, anhydrates, and respective calcining temperatures[36] . . . 15
11 Specification of chalk . . . 16
12 Coded and Actual levels of the variables for CCD of the RSM . . . 26
13 Experimental design setup using design expert CCD . . . 27
14 Physical characterization of the raw materials . . . 28
15 Compositional analysis of Kentry gypsum . . . 28
16 Compositional analysis of Kentry gypsum after crushed . . . 28
17 Compositional analysis of Filiklik gypsum . . . 29
18 Compositional analysis of lower and upper Yogef gypsum . . . 29
19 Comparison of compositional analyses for the raw materials . . . 29
20 Statistical results for actual and experimental . . . 31
21 Optimization criteria used in this study . . . 32
22 Analysis of variance table for improved model for Bulk Density . . . 34
23 Analysis of variance table for improved model for Breaking Strength . . . 35
24 Optimum solutions obtained depending on the desirability . . . 42
25 Validation test results . . . 43
26 The comparison of this result with the literature review . . . 43
27 Demand and supply of chalk in Ethiopia (2000 - 2013) . . . 45
28 Purchased equipment cost . . . 55
29 Fixed Capital Investment Cost . . . 55
30 Raw and auxiliary materials, Utilities cost and Operating Labor Cost . . . 56
31 Experimental Design Run Sheet . . . 65
32 Design Summary of Designs Expert . . . 65
33 Regression Coefficient of 2FI and Significance for Bulk Density . . . 66
34 Regression Coefficient of quadratic and Significance for Breaking Strength 66

LIST OF FIGURES

1 Schematic illustration of the layer structure of gypsum [7] 4
2 Section of the crystal structure of gypsum; the horizontal dotted line shows the plane of cleavage through the water molecules[9] 5
3 Production Chalk in Ethiopia 8
4 The process flow diagram of POP production line[29] 11
5 Plaster production process 14
6 The process flow diagram of batch- type manual chalk production line[47] 17
7 The process flow diagram of automated continuous-type production line . 18
8 Flowchart Outlining the Analysis Steps Undertaken 25
9 Plots of Residuals for a) bulk density b) breaking strength 36
10 Plot of the actual and predicted values for a) bulk density b) breaking strength 36
11 Cubic representation of interaction effects of the three factors on bulk density 39
12 Response surface plots for bulk density as a function of three factors 39
13 Cubic representation of interaction effects of the three factors on breaking strength 40
14 Response surface plots for breaking strength as a function of the three factors 41
15 Material balance for dryer 48
16 Material balance for moulder 48
17 Material balance for mixer 49
18 Material balance for Pulverizer 49
19 Material balance for kettle 50
20 Material balance for grinding mill 50
21 Material balance for secondary crusher 51
22 Material balance for jaw crusher 51
23 Energy Balance on Kettle Calciner 52
24 Energy Balance on Dryer 52
25 Perturbation plots for (i) bulk density and (ii) breaking strength 66
26 Direct effect of the three factors on the bulk density 66
27 Direct effect of the three factors on the breaking strength 67
28 Effect of two factor interactions on bulk density 67
29 Effect of two factor interactions on breaking strength 67
30 Contour plots for bulk density 68
31 Contour plots for breaking strength 68

ABSTRACT

The study was conducted with objective that find the optimum value of the gypsum processing parameters for the production of improved quality chalk using locally available raw materials. Gypsum rocks with size less than 20cm in diameter and 30kg total samples were collected from Educational Material Production and Distribution Enterprise center; the collected samples were taken and the laboratory test processed. Experiments have been conducted with varying influential process parameters to got different responses. Second degree polynomial models for the responses as a function of the independent variables were generated and optimum conditions were determined using Central Composite Face Centered Design of Response Surface Methodology.The data were analyzed to determine optimum conditions for testing responses parameters particle size (90,70 and 45 μm), calcinations temperature (135, 140 and 145 ^oC) and moisture content (5, 6, 7%); for testing the responses bulk density((1.121 - 1.201g/cm^3) and breaking strength (10 - 20N/cm). The optimum value 1.1445g/cm^3 bulk density of and 13.85N/cm breaking strength were achieved at the optimum process parameters 85.55μm particle size, 137 ^oC calcinations temperature and 6.25% moisture content. Depending on the optimum conditions determined experimentally and the existing condition of the plant, chalk production plant process flow sheet has been proposed and the equipments have been designed in which case most of the equipments can be easily fabricated locally. Finally, the plant is financially evaluated and its initial investment cost will be recovered fully within only three years, which makes the plant viable.

Key words: *Gypsum, Plaster of Paris, Response Surface Methodology, Chalk*

1 INTRODUCTION

1.1 Background

Gypsum is sedimentary rock which settled through the evaporation of seawater trapped in lagoons of subsoil and is usually found in mountains. It was formed when seawater evaporated on the earth's surface. Before that, sea levels were much higher than they are today. As climate changed and sea levels lowered, seawater on what is now dry land evaporated, leaving gypsum deposits behind. It is the only natural substance that can be restored to its original rock-like state by the addition of water alone [1].

Ethiopia is rich with natural resources including gypsum but has no enough gypsum plaster industry for construction purposes while it is abundantly used all over the world. According to the available literature, gypsum plaster and its products are energy saving, fire resistant, good insulating, cost effective and possess excellent thermal, acoustical and aesthetic properties [2].

Chalk was first formed into sticks for the convenience of artists. The method was to grind natural chalk to a fine powder, then add water, clay as a binder, and various dry colors. The resultant putty was then rolled into cylinders and dried. Although impurities produce natural chalk in many colors, when artists made their own chalk, they usually added pigments to render these colors more vivid. For example, Carbon was used to enhance black, and ferric oxide (Fe_2O_3) created a more vivid red [2].

An important change in the nature of classroom chalk paralleled a change in chalkboards. Blackboards used to be black, because they were made from true slate. Manufacturers began to fashion chalkboards from synthetic materials during the twentieth century and they chose the color green, arguing that it was easier on the eyes. Yellow became the preferred color for chalk[3].

The plaster of Paris and chalk manufacturing plant was established in 1970 year under the education office of the Shoa Province and rehabilitated in 1982. Although significant changes have been made to improve the production capacity and the quality of the output, there remains much to be done in terms of quality and quantity in order to satisfy the country plan [4].

1.2 Statement of the Problem

Ethiopia is one of the developing countries with a big rich resources, high population and containing many of the under employed adults [4]. The country has been implementing different strategies to bring these unemployed people to employ. Diversification of jobs, increasing the availability of different product through domestic production, and

encouraging the production of high yielding products in different regions of the country are some of such strategies.

It is obvious that the Ethiopian government has launched the growth and transformation plan. To meet this plans, the country requires educationally transformed generations. The education process at all levels needs educational supportive materials, one of these supportive materials is writing board chalk.

Even if the technology of producing chalk is already there in the country, the existing product do not favor the quality, performance and features. Therefore, characterization of the scale helps to direct attention towards the component(s) responsible for the formation of the optimizing and to assess the source of the raw materials. Even though, the raw materials is locally available; our country is importing chalk from different countries that incur foreign currency. In the recent year, the country started to use the locally available raw materials but still the quality is not as such good. It is known that educational sectors in the country, be it private or governmental needs a good quality of writing chalk.

The major problems of chalks observed during use are:

- too dry and difficult to write with
- fragmentsizes easily and turns to dust or powder
- difficult to wipe from the board
- emits a shrill, piercing and scratching sound when applied on the blackboard
- not uniform or consistent in quality
- found broken pieces in the package

Due to these shortcomings, teachers and others who use the chalk are dissatisfied. This is due to use of poor quality of raw materials and processing parameters, and equipment design. This thesis is dedicated to produce improved quality chalk using locally available raw materials; to investigate the effect of different process parameters such as temperature, types of raw materials, particle size, and moisture content and processing time. The thesis will also to determine the optimum process parameters for high quality product.

1.3 Objective

1.3.1 General Objective

The general objective of this thesis work was to optimize the gypsum processing parameters for the production of improved quality chalk using locally available raw materials.

1.3.2 Specific Objectives

The specific objectives of the thesis were the following:

- To study compositional analysis of raw materials.
- To optimize the processing parameters.
- To produce high quality of gypsum powder.
- To minimize the dust of the product.
- To test the quality of product.
- To study the technical and economic feasibility.

1.4 Significance of the Study

Finding of the research adds to the knowledge and understanding of the production of chalk using locally available raw materials in spite of importing. It can expand the technology in the country and help the people to get quality chalk in a simple way [3].
This study should be significant in the sense that:

- Produce low cost chalk by using locally available raw materials.
- Investigate the potential of gypsum under different process conditions.
- Provide useful knowledge on factors that might have impact on quality of chalk.

2 LITERATURE REVIEW

2.1 The Structure of Gypsum System

Gypsum is a soft sulfate mineral composed of calcium sulfate dihydrate, with the chemical formula CaSO_4.2H_2O and the most abundant sulphate mineral. It is water-soluble and forms by evaporation or because of the mineral anhydrite being hydrated. These unique evaporative and soluble properties are two factors, among many others that have led to the unlikely formation of the largest gypsum sand dune field in the world [5]. The presence of gypsum on or just below the surface of the ground is often indicated by changes in vegetation; some plants thrive on gypsum-rich soil whereas others are not at all tolerant of those conditions [6].

As shown in figure 1, the gypsum crystal, which has a monoclinic structure, is made up of layers of calcium atoms and sulphate groups, separated by sheets of water molecules. Ionic bonding forms the CaSO_4 layer, while the water layer is formed by hydrogen bonding. This structure provides the explanation for the ease with which crystals of selenite can be split into layers (cleavage), because the plane of cleavage parallels the planes containing the water molecules[7].

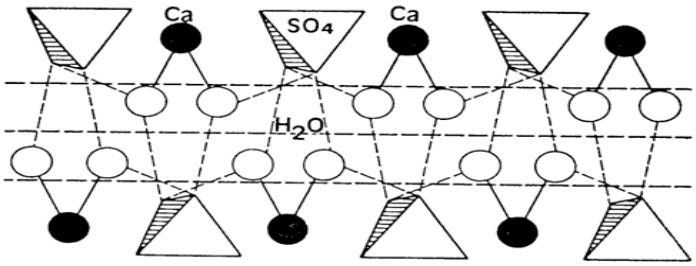


Figure 1: Schematic illustration of the layer structure of gypsum [7]

As shown in figure 2, each calcium ion is coordinated by six oxygen atoms of the sulphate group and by two water molecules. A three dimensional X-ray structural analysis conducted by [8] has revealed that significant departure of the CaSO_4 ion from tetrahedral symmetry occurs. The tetrahedral symmetry increases after removal of water molecules, the S-O distance becomes uniform in natural anhydrous calcium sulphate [9].

2.2 Geological Occurrence and Abundance of Gypsum

Gypsum is a mineral available all over the world but the main sources are the United States, Canada, France, Italy and Britain. It forms as an evaporate mineral and as a hydration product of anhydrite. It occurs in extensive masses of great thickness, in association with limestone and shale and in evaporative deposits; it is very soft and this is one of the easiest methods of distinguishing it from other minerals such as limestone. Its deposits

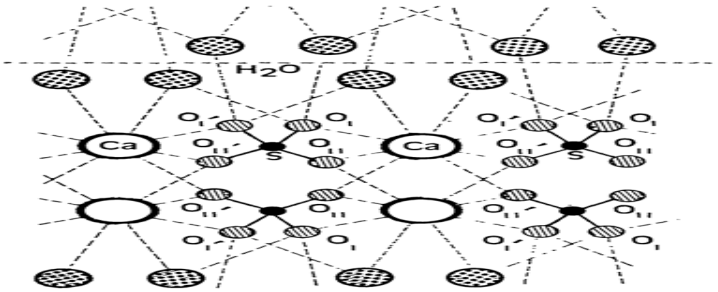


Figure 2: Section of the crystal structure of gypsum; the horizontal dotted line shows the plane of cleavage through the water molecules[9]

are found in water marine evaporates, caves where the air is dry enough for deposits to form, areas where the earth's crust has opened and occasionally in oxidized areas of sulfide deposits. It can also be prepared by precipitation from solutions of calcium salts and sulphate at room temperature or by allowing plaster to set in water[10].

Gypsum is found in mineral and rock form. As a mineral, it can form very pretty and sometimes extremely large and crystals. As a rock, it is a sedimentary rock typically found in thick beds or layers. It forms in lagoons where oceans waters high in calcium and sulfate content can slowly evaporate and be regularly replenished with new sources of water. The result is the accumulation of large beds of sedimentary gypsum. Because it is deposited in this environment, it is common for gypsum to be associated with rock salt and sulfur deposits [11].

2.3 Occurrence of Gypsum in Ethiopia

In Ethiopia, gypsum is formed by the hydration of anhydrite at or near surface, but usually passes into anhydrite below 40-50 meters, although this varies according to local geological conditions. In nature gypsum occurs as beds or nodular masses up to a few meters thick and is the products of the evaporation of seawater[12].

Gypsum has a history of usefulness to man dating back several thousand years. The literature review was referred to compare the three gypsum occurrences. These are Derba Mugher site, Filiklik (Blue Nile Gorge) and Yogef (Jimma River) valley. Gypsum resources are known to occur in Kentry, Filiklik, Blue Nile gorge, Yogef and Jimma River. These gypsum occurrences are mainly associated with the Mesozoic sedimentary rocks and encountered as intercalations with calcareous strata. To determine commercially exploitable gypsum resources that are suitable as a raw material for POP and chalk production, the aforementioned three gypsum occurrences were assessed and evaluated hereunder regards to location and access, mode of occurrence, quality and resource (reserve) assessments[13].

[a] Kentry Gypsum Occurrences: There are two gypsum beds, having thickness of 4m and 12m, respectively with a 4m intercalation of sandstone in between. Kentry is

geographically located North West of Addis Ababa. The chemical analyses of this gypsum are (25.56 - 38.54%)CaO, (25.06 - 54%) SO_3 and (9.09 - 18.08%) H_2O [14].

[b] Filiklik (Blue Nile Gorge) Gypsum Occurrence: Filiklik gypsum potential target area is geographically situated North West of Addis Ababa. This can be reached by 202 km asphalt road, along the way through Addis Ababa-Debremarkos highway. The potential of target area of gypsum that is investigated at prospection level is situated about 500 m west of the mining area of gypsum to the left of the main road to Abay Bridge. The gypsum occurrences are morphologically hill forming. The chemical analysis of this gypsum are: (38.30-36.24%) CaO, (51.38-48.78%) SO_3 and (10.35-11.94%) H_2O [14].

[c] Yogef Gypsum Occurrence: The Yegof gypsum deposit occurs in Jema valley near the confluence of Jema and Bedrsina rivers situated North of Addis Ababa. The gypsum is cliff forming with sharp edges, sacrificially showing rill marks.

Table 1: Compositions Yogef gypsum occurrence[15]

Description	CaO(%)	SO_3 (%)	H_2O (%)	LOI(%)	SiO_2(%)	Fe_2O_3(%)	AlO_3(%)
Lower gypsum bed	28.55-31.75	43.23-45.94	14.63-17.34	5.00-5.97	0.70-1.00	0.09-0.15	0.08-0.14
Upper gypsum bed bed	0.12-31.22	4.68-45.76	17.97-18.89	3.5-4.21	0.65-0.82	0.07-0.10	0.09-0.12

According to different literature reviews, gypsum rock has the following compositions of 32.6% CaO (Lime), 46.5% SO_3 (Sulphate) and 20.9% H_2O (water) when very pure. Therefore, the average chemical compositions of gypsum samples collected from the aforementioned occurrences should be evaluated and compared with these ranges of values[16].

According to the information from Ministry of Mining the present annual consumption of gypsum rock (raw material), the existing plant uses the amount of 1,000 - 1,500 tons for POP and chalk production purposes. Therefore, the occurrences of the gypsum rock in the above-mentioned localities shall be future potential resources for plaster of Paris and chalk production, including other small and medium scale gypsum processing plants[17].

2.4 Production of POP and Chalk in Ethiopia

2.4.1 Production of Plaster of Paris

In Ethiopia POP had been producing and marketing since 1986 year and the productions of data spanning the period 2007-2013 are shown in the Table 2. As it can be seen from the table, the production of POP was increasing from year to year.

Table 2: Production of POP in Ethiopia in consecutive ten years

Year (G.C)	2007	2008	2009	2010	2011	2012	2013
Quantity Production(Ton)	438.5	650.2	559.8	944.2	1058.23	1263.45	1511.87

Sources: EMPDE

Since the bulk of demand for POP is not met through local production with the quality and quantity wanted, it imported from different countries for various purposes. The POP imported from overseas is generally of higher quality and fineness and it is mainly used for medical (bone casting), ornamental and industrial uses. An enterprise AJECA is known to import gypsum from Jordan for sales to the construction market.

Table 3 shows that the imported of Plaster of Paris during the period under consideration is characterized by high fluctuations. The imported data ranges from 4.5 tons and 6.5 tons in the year 2012 and year 2013 respectively to 108.6 tons in 2009. In the remaining years, it ranged from the lowest 15.7 tons in 2011 to the highest 58.5 tons in 2008. The average imported of POP of the last four years was about 75 tons, which is not that much significant compared to the locally produced Plaster of Paris in the recent years.

Table 3: Imports of Plaster of Paris

Year (G.C)	2007	2008	2009	2010	2011	2012	2013
Quantity Imported (Ton)	23.3	58.5	108.6	37.4	15.7	6.4	4.5

Source: Ethiopian Revenues and Customs Authority

2.4.2 Production of Chalk

Chalk is used in school and other learning establishments to write on the board. The demand for chalk is high and this has made its production a goldmine. Common blackboard chalk, which was traditionally made from natural chalk, today is made from gypsum. Blackboard chalk is made into sticks and used to draw on blackboards and other rough surfaces. EMPDE had remained to be the sole domestic producer of chalk in Ethiopia for many years. Other producers of chalk came into market only about five years ago, but their number and capacity is growing from time to time [18].

Table 4: Production of Chalk in Ethiopia in consecutive five years

Year (G.C)	Production (Gross)	Production (kg)
2009	263,368	184,358
2010	313,000	219,100
2011	361,364	252,955
2012	368,296	257,807
2013	388,416	271,891

Source: FDRE Ministry of education

To convert from kilograms to gross, one gross is equal to 700 gram and one piece of chalk is 6.7 gram. As the data showed in Table 4 and Figure 3, the capacity of the product was increasing from time to time.

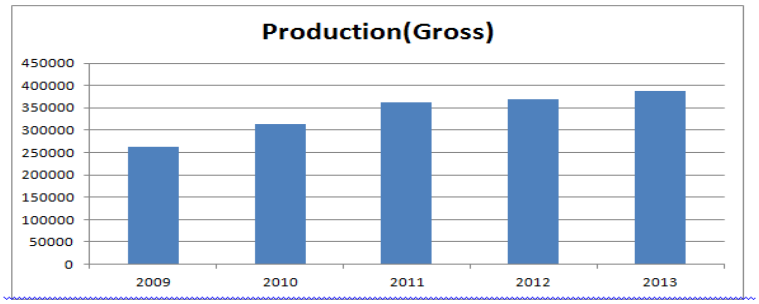


Figure 3: Production Chalk in Ethiopia

The minimum and maximum productions of chalk in the past five years were 263,368 gross in 2009 and 388,416 gross in 2013 respectively. The average production during five years was 32,5892 gross and the corresponding average annual growth of production was 3.2%. This is because of the expansion of school in the country very rapidly. On the other hand, chalk was imported into the country from France, India, UK, Germany, Taiwan, Thailand and Netherlands for many years.

Table 5: Quantity of Chalk Imported

Year (G.C)	Quantity Imported (Gross)	Quantity Imported (kg)
2009	366,925	35,401
2010	404,488	39,025
2011	231,478	22,333
2012	225,676	22,756
2013	198,979	20,064

Source: Customs and Excise Tax Authority, External Trade Statistic, Annual Issues

2.5 The Physico-Chemical properties of Gypsum

Characteristics of gypsum include a low hardness level, perfect cleavage and a monoclinic crystal system of vectors with unequal lengths. Gypsum is a mineral with thick and fine tabular type crystals, large crystals being very rare, crystals are flexible but not elastic, which means that you can bend but they do not spring back, they stay bent. The aggregates are compacting granular, foliated and fibrous. It is a soft mineral and a fingernail can scratch its crystals, which is about the only test needed to distinguish it from other minerals that have a similar appearance. The color of gypsum when reduced to powder is white. The specific gravity of gypsum, i. e., its weight as compared with pure water, is 2.3[19].

Identification: gypsum is the most common sulfate mineral. It is common to find gypsum with calcite, sulfur, halite and anhydrite in layers of sedimentary deposits[20].

Color: Pure gypsum is a white-to-transparent mineral, though impurities can give the

mineral a yellow, grey, red, brown, pink or colorless[21].

Shape: gypsum forms as granular masses, fibrous masses, tabular shapes and prisms. Twin also knows gypsum, to form mirror image crystals on the same level. It is classified under the monoclinic crystal system [22].

Hardness and breakage: minerals are rated using the Moh's scale of hardness. The hardness of gypsum varies from 1.5 to 2.0, the crystalline form being taken as the standard for the second degree in the scale of hardness of minerals[23].

Conductivity: It is a poor conductor of heat, which makes it useful in wall board and drywall which makes it a good insulator[24].

Chemical Formula: Its chemical name is calcium sulfate dehydrate, and its chemical formula is Ca$SO_4.2H_2O$ which means that every dry gypsum molecule has trapped within itself, two loosely attached molecules of water that can be driven off simply by heating it up beyond the boiling point of water[19].

Table 6: Physical and chemical properties of gypsum

Characteristics	Properties	Descriptions
Class	Sulfates	35,401
Chemistry	Ca$SO_4.2H_2O$	It has oxygen, calcium, sulfur, and hydrogen in it.
Crystal System	-	Monoclinic - prismatic
Crystal Habits	-	Include the tabular, bladed or blocky crystals with a slanted parallelogram outline.
Fracture	Conchoidal and splintery	This mineral does not break , into pieces often but when it does it does not do it evenly. The splinters would be smooth.
Hardness	2 Moh's scale	It is so soft that a fingernail can scratch it.
Specific Gravity	2.3	It is a very light stone.
Luster	Vitreous to pearly	When light shines on it, it looks glassy or pearly.
Streak	White	When the stone is rubbed on a white plate, it leaves a white powder.
Cleavage	Good in one direction	If you hit it with a hammer, it would break into layers going in one direction.
Field Indicators	-	Are crystal habit, flexible crystals, cleavage and hardness.
Associated Minerals	-	borax, calcite, halite, pyrite, sulfur and others

(Source: Wikipedia encyclopedia)

Mineral gypsum exists in variety different forms[25]:

- Selenite - Selenite is composed of well-developed crystals, is colorless and transparent, and has a pearl like luster.

- Alabaster - Pure alabaster is a fine-grained massive material which is white and compact. It is commonly carved and polished for statues and ornaments.
- Satin Spar - Satin Spar is a fine translucent variety composed of monoclinic crystals. It is valued for ornaments and jewellery.

2.6 Technology Selection and Production Process of POP

2.6.1 Size Reduction Process

Gypsum rock from the store is transported by a loader and fed to the hopper, then conveyed by a feeder and belt conveyor to the jaw crusher. This is passing through two processes: jaw crusher and small crusher. Primary crushing crush gypsum stone into smaller particles; the general control of particle size is less than 10cm. The larger particles will have re-crushed and smaller particles will be convey by the belt conveyor into the secondary crushing with a hammer crusher machine to control the particle diameter less than 28mm and then transfer by the bucket elevator into the grinding mill[26].

Secondary crushers that work at higher speeds, producing pebbles, accomplish secondary crushing. The crushed gypsum is stored in the hopper from where it is conveyed using a belt conveyor and fed to the hammer mill for further grinding; the ground gypsum is discharged to the hopper and further transported by screw conveyor and bucket elevator to the storage silo. The finely ground gypsum ore is known as land plaster[27].

2.6.2 Kettle Calcinating

There are two distinct types of kettle calcinating technologies, namely; continuous and batch kettle calcinating. However, the only feasible calcinations for small-scale plant is batch kettle calcinations. The land plaster is transported by screw conveyor and bucket elevator and fed to the kettle calciner where it is heated to remove three-quarters of the chemically bound water to form POP. Calcinations occurs approximately 130 to 150^oC and the time taken for calcinations is about two and half hours. During the process of calcinations, the gypsum loses 75% of its moisture thereby making it hydroscopic[28].

There are different types of calcinations necessary to produce POP, but in this paper only the kettle calcinations is used. Because kettle is simple to design and used to hold up to 15 kuntals per batch. Roller mills prepare raw gypsum rock for introduction into calcinating kettles by simultaneously removing the surface moisture. The kettles efficiently remove chemically bound water from the raw gypsum by heating and agitating it in a carefully controlled process. The resulting product, homogenous calcinated stucco is eminently suitable for use either in manufacturing wallboard or in plaster applications [30].

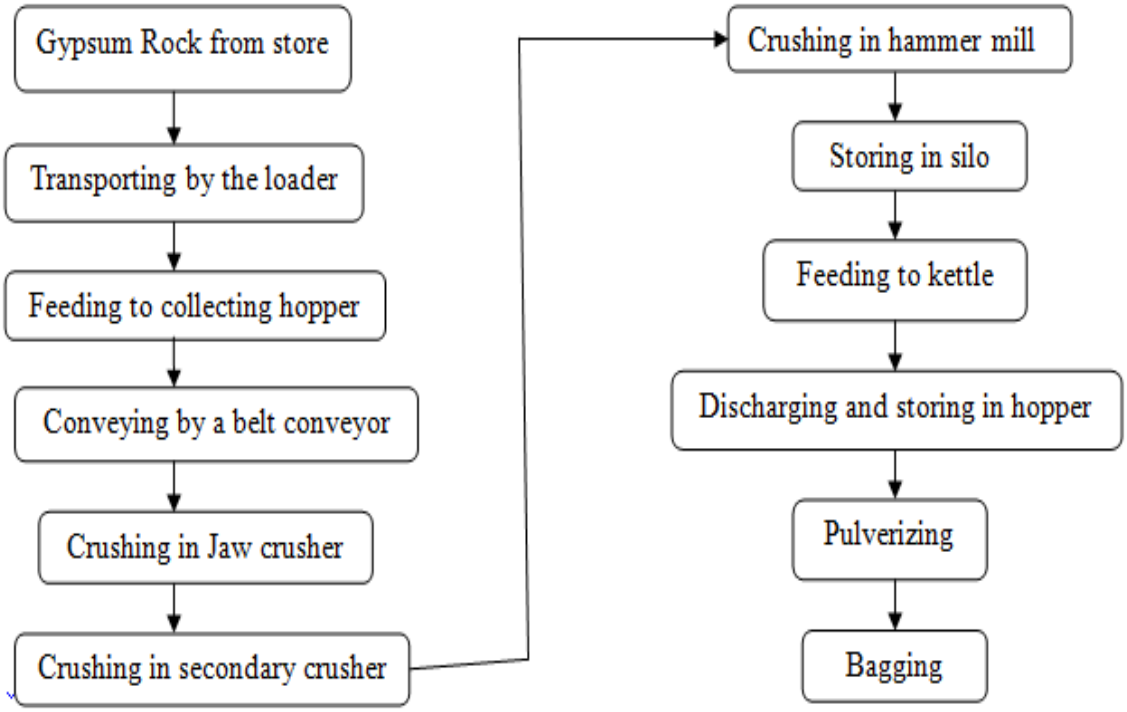


Figure 4: The process flow diagram of POP production line[29]

In the kettle calciner, the plaster is indirectly heated by hot combustion gas passed through flues in the kettle and the product is discharged to hopper located below the kettle. The plaster is conveyed by screw conveyor and put into the hopper for feeding into the pulverizer. The plaster is further ground in pulverizer and transferred to storage silo by screw conveyor and bucket elevator. Packing of POP using plastic bags is carried out at the bottom of the storage silo [31].

2.6.3 Plaster of Paris (POP)

The calcium sulphate dihydrate becomes commercially useful substance by calcinating (i.e. driving out the chemically bonded water) using heat and then grinding it to the required grain size (or fineness)[30]. Chemically, Plaster of Paris is calcium sulphate having molecular formula $CaSO_4.1/2H_2O$. It is a white hygroscopic powder very slightly soluble in water particularly at high temperature. The principal of POP manufacturing is to convert gypsum, dehydrate form of Calcium Sulphate to POP, hemi-hydrate of Calcium sulfate by controlled heating. Hence, production of POP involves dihydration of gypsum and preparing it in usable form (powder)[31].

Based on end use application and heats of hydration two types of POP are produced namely, Alpha and Beta-type POP. Alpha-type is characterized by crystalline nature while Beta-type of is characterized by amorphous nature. After vigorous mixing of the hemihydrates with water, a typical beta hemihydrates plaster require between 75 to 100ml of water per 100g of plaster to give pourable slurry. A typical alpha hemihydrates, on the other hand require only 28 to 45ml of water to give pourable slurry with 100g of plas-

ter[32].

- Calcinations under atmospheric pressure to produce Beta plaster, and
- Calcinations under elevated pressure to produce Alpha plaster. Each category of calcinations of gypsum into plaster was described briefly as follows.

Beta Plaster: Beta plaster presents micro-crystals that yield high porosity and average low mechanical strength only when cast with water, as they need high excess of water in slurry (typically water quantity will be 80% of plaster quantity). The type of plaster required for manufacturing chalk is the beta plaster[33].
Alpha Plaster: Alpha plaster presents well formed crystals that will yield low porosity and high mechanical strength when cast with water, as they need high excess of water in slurry (typically water quantity will be 33% of plaster quantity)[34].

Table 7: Comparison of alpha and beta plaster

Properties	Alpha process	Beta Process
Mechanical strength	Higher	Lower
Crystals	Larger	Smaller
Porosity	Lower	Higher
Specific surface	Lower	Higher
Water demands	Lower	Higher
Density	Higher	Lower
Energy	Higher	Lower
Heat reaction	Larger	Smaller
Calcinations Temperature	Up to 150^oC	Up to 300^oC
Pressures	Higher than atmospheric pressures	At atmospheric pressure

The hemihydrates are probably so named because of the very large deposit of pure gypsum found beneath Paris. It can be mixed with water, spread and cast into different forms and sizes. Being a hygroscopic material (i.e. containing water), calcium sulphate dihydrate undergoes chemical decomposition reactions when heated and the water of crystallization is driven off some or all of the water of crystallization. POP is a chemical having the notable property of setting and subsequent hardening when mixed with water[35].
The chemical endothermic reaction is:

$$CaSO_4.2H_2O + \text{Heat} \longrightarrow CaSO_4.\tfrac{1}{2}\,H_2O + 1.5H_2O$$

During this reaction, gypsum dihydrate to calcium sulphate hemihydrates (or POP) and 75% of the chemically combined water is released. With further increase in the temperature, the remainder 25% of the chemically bound water.

$$\text{CaS}O_4.2H_2O + \text{Heat} \longmapsto CaSO_4 + 2H_2O$$

Rehydration
If the powder is mixed with water to form a slurry or paste, it dry and set rock hard. As the plaster and water, mixture dries, water is chemically recombine with the hemihydrates

gypsum and the material revert to the original composition of gypsum. The basic rehydration reaction is the reverse of calcinations. POP consists of a fine, white powder and calcium sulfate hemihydrates that hardens when moistened and allowed to dry [36]. To convert the reaction above by mixing gypsum product (powder) with water:

$$\text{CaS}O_4.1/2H_2O + 3/2\ H_2O \longmapsto \text{CaSO4.2}H_2O + \text{Heat}$$

When the POP is mixed with water to a pourable state and allowed to set; it reverts back to a solid mass. This process is called recrystallization. The material heats and expands until it reaches the chemical composition of natural gypsum-containing two molecules of water. Certain modifiers can be added to the plaster to accelerate or retard the setting process [37]. Generally, the following reaction can take place in gypsum powder production:

$$CaSO_4.2H_2O\text{(s)} \longmapsto CaSO_4.1/2H_2O\text{(s)} + 3/2\ H_2O\ \text{(L)}$$
$$CaSO_4.2H_2O\text{(s)} \longmapsto CaSO_4\ \text{(s)} + 2H_2O\ \text{(L)}$$
$$CaSO_4.1/2H_2O\text{(s)} \longmapsto CaSO_4\ \text{(s)} + 1/2\ H_2O\ \text{(L)}$$
$$CaSO_4\ \text{(s)} + 1/2H_2O\ \text{(L)} \longmapsto CaSO_4.1/2H_2O\text{(s) (slow)}$$
$$CaSO_4.1/2H_2O\text{(s)} + 3/2H_2O\text{(L)} \longmapsto CaSO_4.2H_2O\text{(s) (fast)}$$

2.7 Gypsum Dehydration Sequence

Plaster is produced from the dihydration of gypsum. Calcium sulphate dihydrate (hereafter referred to as dihydrate) dihydrate on heating to calcium sulphate hemihydrates (hereafter referred to as hemihydrates). The β-hemihydrates is produced by dry calcinations and is the main constituent of plaster. The α-hemihydrates is produced by wet calcinations at elevated pressures or atmospheric pressure with the addition of acid or aqueous salt solutions. Hemihydrates on heating dehydrate further to calcium sulphate anhydrite (hereafter referred to as anhydrite) [38].

The important physical properties of the different calcium sulphate phases were shown in Table 9. Calcium sulphate hemihydrates occur in α and β forms. Plaster consists on the whole of the β-hemihydrates form, which is made up of flaky rugged secondary particles. The α-hemihydrates consists of compact, well formed, transparent, large primary particles. The properties of hemihydrates can lie anywhere between those of the two end members depending upon the water partial pressure when formed[39].

Table 8: Phases of gypsums

Calcium Sulphate	Chemical Formula	Abbreviations
Dihydrate	$CaSO_4.2H_2O$	D
Hemihydrates	$CaSO_4.1/2H_2O$	H
Anhydrite III	$CaSO_4$	AIII
Anhydrite II	$CaSO_4$	AII
Anhydrite I	$CaSO_4$	AI

At high temperatures, dihydrate can be dihydrated directly to anhydrite. Anhydrite exists in 3 forms, AIII, AII and AI depending upon the temperature of reaction. AIII is meta-stable at ambient conditions and will readily rehydrate to hemihydrates in the presence of water vapor. AII is stable at ambient conditions and AI is not stable below $700^{o}C$[40].

Factors affecting the rate of dehydration
It has been found that the rate of the dehydration reaction increases with[41]:

- Increasing temperature
- Decreasing particle size
- Decreasing water vapor pressure
- Decreasing air pressure

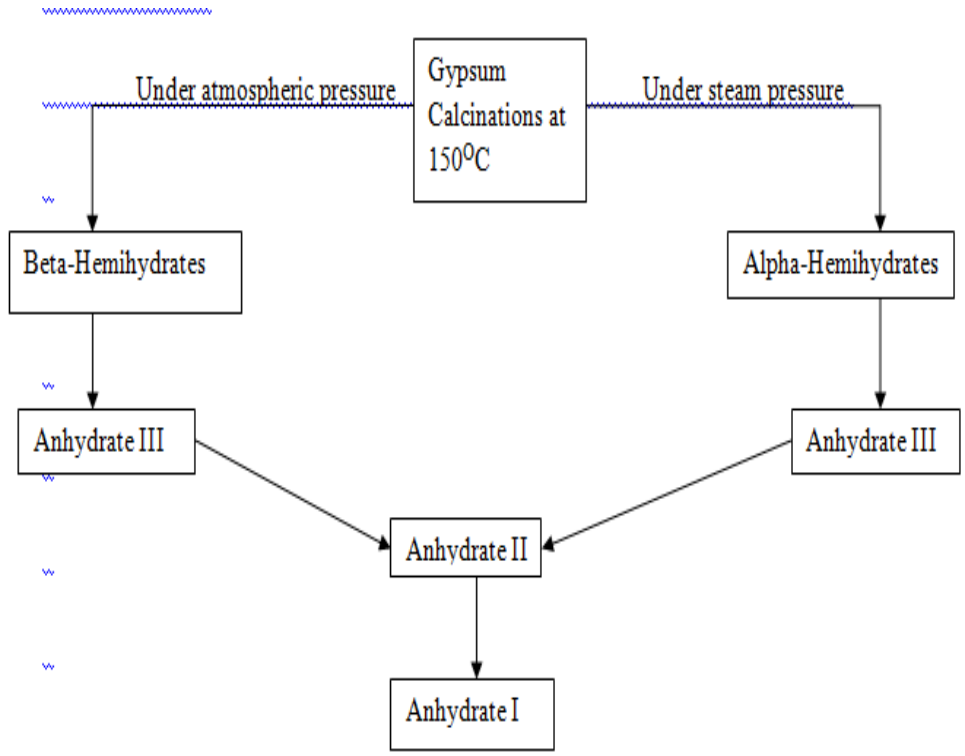


Figure 5: Plaster production process

For the plaster stage, various types of gypsum are produced by differentiating the temperature of the calcining process. Higher temperatures lead in general to a lower reaction with water[42]. As the temperature is increased, the reaction rate increases and there is a greater possibility of the formation of the undesirable anhydrite phases. Gypsum, hemi and anhydrite can co-exist within a single grain of gypsum, with the thickness of each successive layer depending upon temperature, rate of heating, particle size, moisture content and possibility of rehydration.

Table 9: Physical properties of gypsum, hemihydrate and anhydrite

Properties	Dihydrate	Hemihydrates	AIII	AII	AI
Formula	$CaSO_4.2H_2O$	$CaSO_4.1/2H_2O$	$CaSO_4$	$CaSO_4$	$CaSO_4$
MW(kg/kmol)	172	145	136	136	136
Crystallization	21Wt%	5.25Wt%	0	0	0
Density, kg/m^3	2,310	α: 2,757, β: 2,619-2,637	2,580	2,930 - 2,970	Not known
Solubility	0.21	α:0.67, β: 0.88	D to H	0.27	-
Hardness, Moh's	1.5	-	-	3-4	Not known
Thermodynamic	<40^oC	Metastable	Metastable	40-700^oC	>700^oC
Forms/Stages	-	2 forms, α,β	-	-	-
Synthesis Conditions	α:<40^oC	α: >40^oC, β:45-200^oC	-	200-700^oC	>700^oC
Production Temp	<40^oC	α: 80-150^oC, β:220-300^oC	-	300-600^oC	Not produced

The dihydration of dihydrate and hemihydrates is strongly influenced by water vapor pressure. If the water vapor pressure is high, enough at a given temperature then the dehydration reaction does not proceed[43].

Table 10: Hemihydrates, anhydrates, and respective calcining temperatures[36]

Name	Chemical Formula	Calcining Temperature
α-hemihydrate	$CaSO_4.1/2H_2O$	130-150^oC (saturated steam atmosphere)
β- hemihydrate	$CaSO_4.1/2H_2O$	220-300^oC (standard atmosphere)
α-Anhydrite III	$CaSO_4$.0,xH_2O	290^oC(saturated steam atmosphere)
β-Anhydrite III	$CaSO_4$.0,xH_2O	110^oC (standard atmosphere)
Anhydrite IIs	$CaSO_4$	300 - 500^oC
Anhydrite IIu	$CaSO_4$	300 - 500^oC
Anhydrite I	$CaSO_4$	>700^oC

2.8 Technology Selection and Production Process of Chalk

At present, the basic difference between different technologies of producing school chalk is in its being automated or manual production. In the manual production, chalk is produced by moulding on 600 pieces moulding machines. Drawing of the chalk is done by pushing the piston using a mechanism assembled on the moulding machine at the bottom of the cylinders manual pushing handle. Drying the chalk is carried out by hot air heated by kerosene/diesel oil[44]. The advantage of manual chalk production plant is that there is not be plant stoppage if one of the machines stops operation, except the dryer. Its disadvantage is that the efficiency and quality is depending on the dedication of the operators.

In the automatic production, chalk is produced by moulding on cylindrical rubber moulds. The moulding is carried out on closed ring chain conveyor and lasts about 12 - 15 minutes. At the end of the round path, the hardened school chalks are taken out by means of automatic draw machine and automatically put on moving trays. The chalks on trays

get into constant and continuous cycle dryer. Drying is carried out by burning the LPG. The dried chalk is automatically filed into carton boxes and packed. The whole plant is controlled by an operative control system[45]. In automated plant, there is no problem on the quality of the product and on its efficiency since each operation is controlled. However, the plant is forced to stop if one of the machines is out of operation by any reason. Besides, running costs of fully automated plant of a continuous type production in

Table 11: Specification of chalk

Physical and chemical properties	Description
Appearance	White crystalline powder
Specific Gravity	1.14
CaO	39.1%
SO_3	53.2%
Fe_2O_3	0.25%
H_2O	5.5%
Heat capacity, Cp	0.9kJ/kg.K
Density of chalk	1.14g/cm^3
Calcinations temperatures	135^{o}C
Calcinations time	(110 - 130) minutes
Molding time	(12 - 15) minutes
Particle size	90μm
Drying time	480 minutes
Drying temperature	120^{o}C
Moisture contents	7%
Mass of 1 piece of chalk	6.7gm
Diameter of 1 piece of chalk	10mm
Length of 1 piece of chalk	80mm
Bulk Density	(1.121 - 1.201)g/cm^3
Breaking Strength	(10 - 20)N/cm

terms of maintenance cost and complexity, cost of fuel, etc are higher than that of manual plant. However, product quality, operation efficiency, unitary machines output, etc. are higher for an automated plant [46].

1. Manual Batch- type production of chalk

Correctly, measured amount of water using a handle is poured into the stirring tank. Then the raw material, calciner is correctly measured on a scale and is poured into the stirring tank. Mixed oil is painted with a brush on the inner side of the mould beforehand. The milky slurry mixture is passed through 90Âţm filter and filled into the mould.

The work from stirring the raw material up to finishing the moulding is done in 60 - 80 seconds. In order to minimize the bubble in the mixture which has been poured into the mould, the moulder is given a slight jar to release the air bubbles from the top. The ratio of mixed oil used as mould releasing agent is 200 liters of kerosene to 3 liters of any oil.

The time to draw out the chalk is approximately 12 - 15 minutes after moulding. When the chalk is ready to be drawn out of the moulding machines, a drawing frame is placed on the top of the moulding frame and the moulding machines is inclined 90 degree, then

the chalk is automatically fall into the drawing frame.

The drawn chalk is taken away, and put in a designated place, arranged in a drying

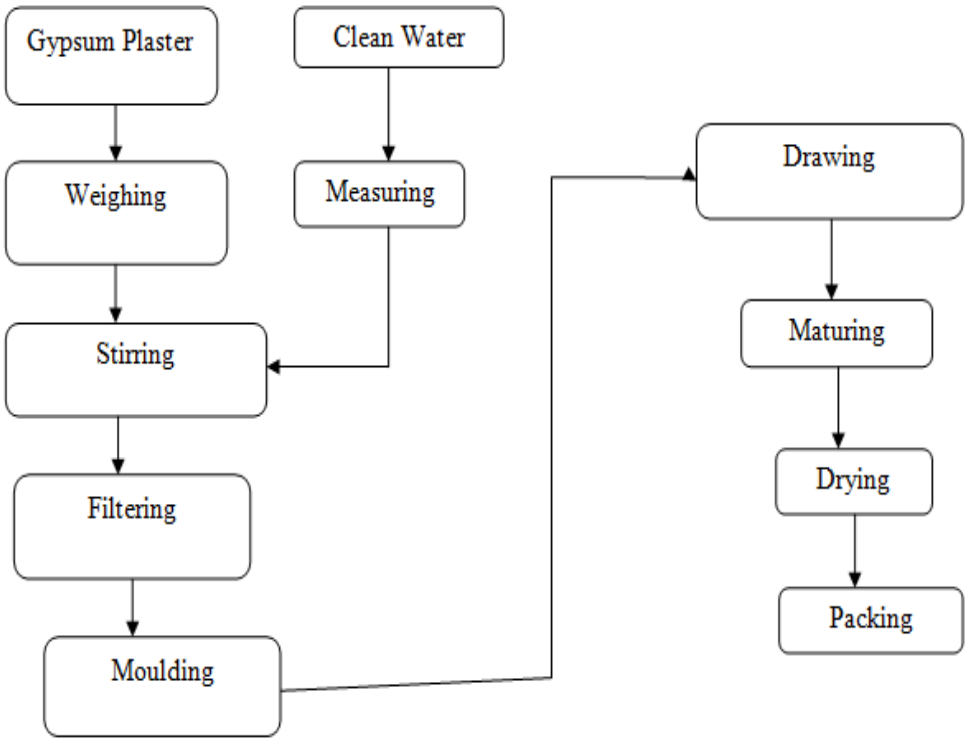


Figure 6: The process flow diagram of batch- type manual chalk production line[47]

frame and then dried in the drier. The dried chalk is then packed in a carton manually. The advantages of using manual batch-type plant are that it is simple to operate with relatively low running costs like cost of maintenance, spare parts and source of power. The main disadvantages are lower unit output of production equipment, especially the moulding machines, stirrers, etc. and inferior product quality[48].

2. Automated Continuous- type production of chalk

The plaster from silo, silo is discharged by screw conveyor and fed into an automatic mixer. Water is simultaneously poured into the automatic mixer with an electronic pump which regulates the water flow rate. The raw materials are measured accurately and mixed in the mixer. The automatic mixer pours the already automatically pre-mixed plaster mixture into special rubber moulds. The moulds have the pre-selected shape according to the type of school chalks and lie on a transportation line composed of mobile cars[49].

After pouring the plaster mixture into the shapes, a closed-ring, linear, horizontal process was started lasting about 14-17 minutes. At the end of the round path, the already hardened school chalk is removed by means of an automatic pattern draw machines and automatically put on other flat moving trays. The chalk on the trays then gets into the constant and continuous cycle dryer. At the dryer output, a selection of the product takes

place by means of an automatic optical system where the damaged product is scrapped while the equality one is automatically placed into 144 unit boxes. Then the boxes are stored in a warehouse.

The automatic continuous-type plant has the advantage of high unit output of equipment and quality final product. High cost of spare parts, maintenance and power are among the disadvantage of the automatic continuous-type production of chalk. However, the advantages of the automated continuous-type production technology overweigh its disadvantages[50].

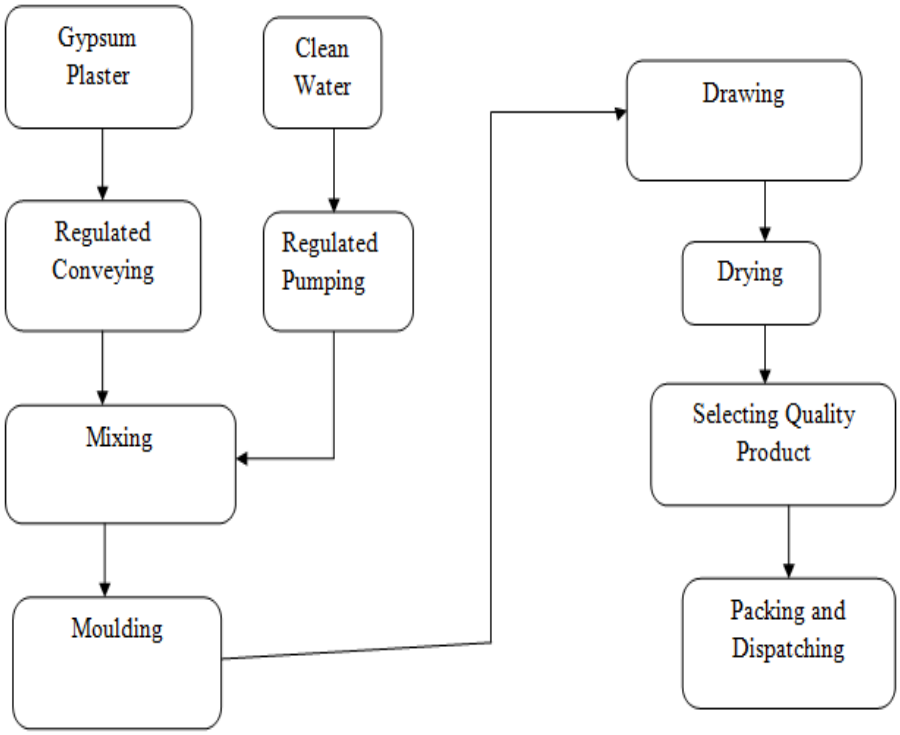


Figure 7: The process flow diagram of automated continuous-type production line

2.9 Different Processing Parameters on Chalk

Functional properties refer to physical and chemical properties of a chalk or chalk component that affect the quality of the product. Parameter processes of chalk production such as particle size, temperature, time and moisture content predict the functional properties of products[50].
The most important dependent variables after processing are the product quality characteristics. Yield strength and bulk density are related to these parameters in different ways. As the size of the particle decreases, the time taken to evaporate the moisture content from the raw materials decrease. This means the moisture content of the gypsum powder evaporate by a minimum temperature. The high dependence of bulk density and yield strength on temperature would reflect its influence on the quality of the product. As temperature increases, the moisture content during calcinations decreases and

the time increases. Therefore, decrease in particle size decreased yield strength and increased bulk density[51].

The properties of gypsum during size reduction process are influenced by the machine variables (e.g. types of the machine and design), the parameter processing variables (e.g. calcinations temperature, calcinations time, moisture contents and particle size) and types of raw materials used. The interaction among all these variables transforms the feed materials leading to the changes in product qualities[52].

2.10 Other Possible Raw Materials to Produce Chalk

There are other possible raw materials in the country to produce chalk. These raw materials are dolomite and calcium carbonate. Dolomite is a common rock-forming mineral. It is a calcium magnesium carbonate with a chemical composition of CaMg $(CO_3)_2$. Chemically dolomite is a double carbonate of calcium and magnesium with a pronounced crystalline structure. However, the problems with these raw materials are that they need maximum temperature for calcinations and have many problems on the environmental[53].

2.11 Application of Gypsum

The most general use of gypsum products is in those structures where their lightweight and fire-resistant qualities make them especially desirable. It is used to retard the setting time of cement (average 3 - 6% added to the clinker), as a fertilizer, as a source of calcium and sulphur and as soil conditioner. It is also used as a substitute for sodium sulphate in glass making. It is inert with good absorbing capabilities. Together with inertness and lack of abrasiveness, some grades of gypsum are sufficiently pure to be used as a filler or diluents in paint, plastics, textiles, food and pharmaceutical industries[54].

Best quality, finely ground white gypsum is used as a base in cold-water paints and in the manufacture of certain cheaper grades of pigments. Plaster of Paris is a basic ingredient of many plastic paints as a hardening agent being added to make the material more durable. In the textile industry, gypsum in its ground form is used in the finishing of cotton and cloth to give weight to a product when a lustrous or glossy surface is required[55].

Calcined gypsum is also widely used in the pottery industry as moulding plaster. In dentistry and orthopedics, gypsum is used as a moulding and casting material. Plaster board or gypsum board, which consists of sheets of slabs of gypsum mixed with fibers, is employed as fire resistant material for walls, ceilings or partitions. In the chemical industries, gypsum is used in the manufacture of sulphuric acid and ammonium sulphate[56].

2.12 Environmental Implications

Gypsum crystal is usually found in conjunction with or near sulfur deposits. If the calcium sulfates in gypsum mixes with the sulfur, then toxic chemicals can be produced, so the gypsum must be mined properly to ensure the substances remain separate. One chief use for gypsum is in drywall used to build houses. Contaminated gypsum inside the walls of a house would jeopardize the health of the environment and surrounding communities[55].

The process of extracting the gypsum from the ground can cause environmental degradation. According to the Global Press Institute, gypsum mines in India leave scars on the landscape. Not only are they unpleasant to look at, but all structure has been removed from the landscape and deposits of gypsum are left exposed to the elements. The lack of rock or vegetation leads to soil erosion and causes sinkholes and landslides. Exposed gypsum dissolves easily in the rain and leaves gaping holes that both contribute to the erosion and pose a physical danger [56].

The product does not create any noise or water pollution. The air pollution has to be continuously monitored. Masks can be used by the workers while making powder (grinding). The production of plaster and plasterboard puts very little pressure on the environment. The final product is non-toxic and wastage and unwanted by-products from the process are minimal. The technology uses gypsum and fuel only and its waste product is mainly gaseous water and carbon dioxide. So, the process is environmentally friendly [57].

3 MATERIALS AND METHODS

3.1 Time and Place of the Study

The study was conducted from October 2013 to October 2014 at Addis Ababa University, Addis Ababa Institute of Technology: school of Chemical and Bio Engineering in Process Engineering laboratory (AAiT) and the results were tested in Geological Surveys of Ethiopia and Food Engineering Laboratory (Hawassa University).

3.2 Materials

The materials used to run the experiments are listed below:

- Jaw crusher, Secondary crusher, Miller and Pulverizer - for size reduction of rock
- Kettle - to evaporate the water contents from gypsum
- Moulds -to shape the mixture
- Scraper - for scraping the surface of the chalk mould.
- Paint Brush - for applying lubricant on the mould before filling it up with POP.
- Lubricants: It could be any cooking oil or coconut mixed with kerosene. Its purpose is for lubricating the chalk mould to prevent the POP from sticking to it.
- Ultramarine Blue and Fe2O3: This is used to give the chalk color but it is optional if one is interested in producing only white chalk.
- Sieves(mesh size of 45μm, 70μm and 95μm Sortmks-3332, PFEUFFR, Germany) with shaker -for size classification
- A Spatula (Mingling stick)
- A weighing Scale (grammometer)
- Fuel - for calcinations
- Tray Dryer - for holding the wet chalk sticks until they dry

3.3 Methods

3.3.1 Source and Preparation of Raw Materials Sample

Gypsum selection and preparation (cleaning and classifying) are the key factors to produce the best plasters and quality chalk. Gypsum rocks with size less than 20cm in diameter and 30kg total samples were collected from EMPDE center, which is located in Addis

Ababa; the collected samples were taken and processed the laboratory test. All other materials such as paints, oils, gasoline, fuels, packaging materials etc. were bought from local market. All samples were transported to Process Engineering laboratory of AAiT for further works within 20 days.

3.3.2 Size Reduction of the Sample

The size of the taken samples was adjusted to less than 10cm in diameter by hammer. In jaw crusher the sample was reduced to a size less than 5cm in diameter and further reduced to a size less than 20mm in hammer mill. The particle size of the sample was further reduced in grinding mill to less than 2mm which increased the amount of fine particles in the powders. It is important to select proper grinding mill with high efficiency and capacity [31]. Finally, the sample was pulverized to a particle size less than 120μm and the optimum size was determined. The sample was sieved using vibrating shaker (Retsch) with set of sieves arranged in descending order 95μm, 70μm and 45μm and this is to investigate the effect of particles size on the quality of chalk [35] .

3.3.3 Calcinations Procedure

The grounded gypsums were calcinated in an oven within a temperature range of (135 - 145^oC) and the calcinations temperature was chosen accordingly for all the samples. The calcinations time was dependent on the working time of machine. When the kettle starts work, it takes more time to evaporate the water content. As the machine stayed on work for two and more batches, the time it takes was decreased. This is because when the machine was very hot, the wanted temperature was arrived in a short time. For each experiment, 500g of the grounded and sieved samples was weighed and put in an oven. These samples was calcinated at different temperatures in the stated range until 70%, 75% and 80% of the moisture content was derived off in the form of vapor. Calcinations temperature was measured by a thermocouple inserted along the length of the kettle calciner [30].

3.3.4 Mixing Procedure

500gm of POP was weighed and mixed with 600ml of cold clean water in bucket and white chalk was produced. 490gm of POP was weighed and mixed with 600ml of clean water and 10gm of any wanted colors and colored chalk was produced which is standard Worldwide [31]. It was mixed properly back and forth motion while turning the container every once in awhile and made sure that the mixture is not watery and at the same time not too thick. This will allow the POP to be evenly incorporated with the water. The sides of the bowl must also be scraped to make sure there are no dry samples sticking to the sides. The mixing was continued until an even consistency achieved. There should be no lumps in the POP mixture. After mixing the POP with water, the resulting slurry must be

used within two to three minutes. POP when mixed with water tends to dry up fast. It is best to have the moulds ready so that the slurry can be poured into them immediately; it cannot be kept; it must be poured into mould otherwise it is wasted [44].

3.3.5 Moulding Preparation

The pieces of the mould should be washed very well with the mixture of oil and kerosene; couple the mould together with the provided treaded rod and make sure they are tightly closed to avoid leakage. The ratio of kerosene to oil mix used as mould realizing agent kerosene to oil is 3:1 litters and painted with a brush on the inner side of the mould beforehand. Pour the solution into the mould; shake the mould and use your hand to guide the solution as you are pouring to ensure that it gets to bottom of the mould and fill it properly. The solution should be poured at the center of the mould then using scraper smear the mixture into mould hole. The slurry mixture remaining on the top surface of the mould frame is wiped off with a metal spatula, and then the top surface of the mould is rapidly finished off with a finishing spatula[45]. It is allowed the solution to stay in the mould for 12 to 15minutes to give the shape of chalk and this is the first stage at which the drying take place. When the chalk is ready to be drawn out of the mould, a drawing frame is placed on the top of mould frame and the drawing stopper is set firmly. The mould is inclined 90 degrees toward the operator and the chalks automatically fall in to the drawing frame. The drawn chalks are then taken away, put in to the designed place, and arranged in trays for drying[44]

3.3.6 Drying Procedure

Now the arranged trays were entered into the dryer and dried at 120oC. Moisture is removed from water-based coatings and adhesives, as well as from the surface and interior of products or substrates. Testing: Laboratory testing was carried out at several production phases to ensure all products meet the strict product specifications before being bagged and shipped.

3.4 Proximate Determination

All raw materials' samples were analyzed in duplicate for their proximate composition according to the following procedures.

3.4.1 Determination of Moisture Content

Moisture content was determined by drying the samples in an oven until a constant weight obtained. The 500 grams of samples and 6grams of the aluminum foil; total of 506 grams were accurately weighed and put in kettle and dried at the 135, 140 and 145^oC

temperatures. The process was repeated until a constant weight obtained [48]. The moisture content was determined using equation. (1).

$$\mathbf{MC}(\%) = \frac{\mathbf{Weightofsamplebeforecalcination} - \mathbf{Weightofsampleaftercalcination}}{\mathbf{Weightofsamplebeforecalcination}} \quad (1)$$

3.4.2 Determination of Bulk Density

To determine the bulk density of the chalk, the chalk was weighed and put in cylinder with clean water and stayed for 3 to 4 minutes. The wet chalk was taken from the water and measured carefully [59]. Repeating this experiment three times and recording the weight, bulk density of the chalk had been calculated as follows in equation (2):

$$\mathbf{BulkDensity}(\mathbf{N/cm^3}) = \frac{\mathbf{MassofWetChalk}(\mathbf{g})}{\mathbf{VolumeofChalk}(\mathbf{cm^3})} \quad (2)$$

3.4.3 Determination of Breaking Strength

The chalks were placed on the sample platform of 1kg load cell with 2 - 6cm specimen TA-XT plus texture analyzer. The load was released vertically to the chalk. The maximum force in Newton (N) required to break the samples exceeding a breaking limit was recorded and the average of three samples was used for calculation. Hardness and breaking strength of chalk was determined as the maximum force offered by chalk during breaking and three point diameters [59]. The breaking strength was calculated using equation (3):

$$\mathbf{BreakingStrength}(\mathbf{N/cm}) = \frac{\mathbf{BreakingForce}(\mathbf{N})}{\mathbf{Distanceb/nloadandchalk}(\mathbf{cm})} \quad (3)$$

3.5 Experimental Design of Process Parameters

Experimental designs nowadays have been regarded as one of the most favorable techniques in covering a large area of practical statistics and obtain unambiguous results with the least expense. An experiment is a series of tests, called runs, in which changes are prepared in the input variables in order to recognize the reasons for changes in the output response [60]. Design of experiments is a powerful technique used for exploring new processes; gaining increased knowledge of the existing processes and optimizing these processes for achieving world class performance and enable designers to determine simultaneously the individual and interactive effects of many factors that could affect the output results in any design [61].

Optimizing refers to improving the performance of a system, a process, or a product in order to obtain the maximum benefit from it. Often engineering experimenters wish to find the conditions under which a certain process attains the optimal results. i.e., by careful design of experiments, they want to determine the levels of the design parameters

at which the response reaches its optimum. The optimum could be either a maximum; a minimum or target responses which is influenced by several independent variables (input variables). One of methodologies for obtaining the optimum results is response surface methodology [62].

Response surface method (RSM), invented by Box and Wilson, is defined as a collection of mathematical and statistical tools or techniques useful for modeling, analyzing and simultaneously solving problems in which a response of interest is influenced by several variables and the objectives is to optimize this response [63].

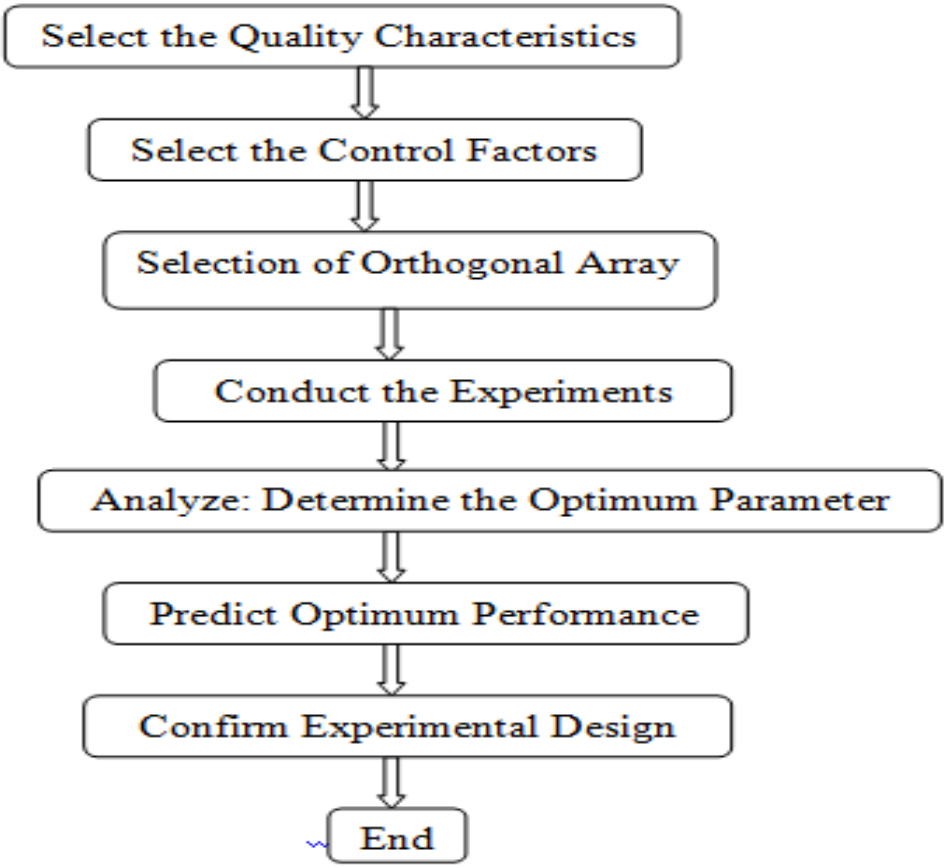


Figure 8: Flowchart Outlining the Analysis Steps Undertaken

It also has important applications in the design, development and formulation of new products, as well as in the improvement of existing product designs. The field of response surface methodology consists of the experimental strategy for exploring the space of the process or independent variables, empirical statistic modeling to develop an appropriate approximating relationship between the responses and process variables, and optimization methods for finding the levels or values of the process variables that produce desirable values of the responses. The main advantage of RSM is the reduced number of experimental runs needed to provide sufficient information for statistically acceptable results [64].

3.6 Factors Affecting the Quality of the Product

Quality of the chalk can be affected by different factors such as particle size, calcinations temperature, moisture content, calcinations time, moulding time and other minor factors. However, none of these factors are equally contributes to the quality of the product. Out of these factors affecting the quality of the product, particle size, calcinations temperature, and moisture content are the most relevant factors due to their significant influence [59]. Central Composite Design (CCD) was used and the data were analyzed with Design-Expert 7.0 program. CCD of RSM was used to determine optimum conditions for testing responses parameters[65]. Therefore, in this work, optimization of process parameters (PS, CT and MC) for testing the responses (bulk density and breaking strength) of the product had been carried out and the influence of process variables on these responses of the product was analyzed.

3.6.1 Experimental Set-up

The setting of the process variables was done based on the chemical proximate composition of the raw materials, different literatures and preliminary tests. These ranges of the factors have been selected by practical experience of the process and then conducting pre-experimentation tests. CCD is well suited for fitting model surface, which usually works well for process optimization because it includes center and axial points [66]. Center points, for which all the factor values are at the zero (or midrange) value. Axial (or star) points, for which all but one factor are set at zero (midrange) and the other factors are set at outer (axial) values [67].

Table 12: Coded and Actual levels of the variables for CCD of the RSM

Factors	**Unit**	**Coded symbol**	**level 1 (-1)**	**Level 2 (0)**	**Level 3 (+1)**
Particle Size	(μm)	A	45	70	95
Calcinations Temperature	^oC	B	135	140	145
Moisture content	%	C	5	6	7

Face centered composite design (FCCD) for three factors (particle size, calcinations temperature and moisture content) and three level combinations with total number of 20 runs were used to conduct the study.

- Number of factors, f = 3
- Run = 2k + 2k + n_c = 23 + 2x3 + 6 = 8 + 6 + 6 = 20 and Replicate, r = 3
- Number of total experiments = 20 * 3 = 60

The study was conducted to optimize the process variables to produce quality chalk by varying the process variables of particle size (45, 70, 95μm), calcinations temperature (135, 140, 145^oC), and moisture content (5, 6 and 7%). The aim of varying these variables

was to get different values of responses (bulk density and breaking strength). The data obtained from the experimental laboratory was analyzed using the software to determine the optimum response variables.

Means comparisons was performed using analysis of variance (ANOVA). Finally, the products were selected which gave a better responses and the validation was done on the selected parameters. Studies were conducted using the RSM in modeling the physical properties as affected by main variables of individual and their interaction effects. The independent and dependent variables were fitted to the second order model equation and examined for the goodness of fit. The behavior of responses variables as a function of regression explanatory variables were represented by RSM using the following equation:

Yi = β_o + β_1A + β_2B + β_3C + β_{12}AB + β_{13}AC + β_{23}BC + $\beta_{11}A^2$ + $\beta_{22}B^2$ + $\beta_{33}C^2$ + ξ

.....(4)

Where: Yi - the response function (i stands for bulk density and breaking strength);

β_o - constant; β_{ij} - regression coefficients (i = 1,2,3 and j = 1,2,3,);

A - Particle size; B - Calcinations temperature; C - Moisture content and; ξ- Random error

Table 13: Experimental design setup using design expert CCD

Run	A:Particle Size	B:Calcinations Temp.	C:Moisture Content
1	45	140	6
2	70	140	6
3	95	135	7
4	70	135	6
5	70	145	7
6	70	145	6
7	95	145	7
8	70	140	7
9	45	145	5
10	70	145	5
11	95	135	5
12	95	145	5
13	95	140	6
14	70	140	5
15	45	145	7
16	45	140	5
17	95	140	7
18	95	145	6
19	45	135	7
20	45	135	5

The resulting data for optimization of quality chalk production from gypsum and the effect of three operating variables were analyzed to generate significant effects with two responses. Numerical optimization techniques of the Design-Expert software's were used for simultaneous optimization of the multiple responses [68].

4 RESULTS AND DISCUSSION

4.1 Characterization of the Raw Materials

4.1.1 Physical Characterization

The raw material collected was first characterized physically in such a way that visual observations, touching and trying to break it. The physical characterization of the raw materials was as in Table 14.

Table 14: Physical characterization of the raw materials

Properties	Color	Density	Breakage
Type/amounts	White	2.3 g/cm^3	1.5 Moh's scale

4.1.2 Chemical Characterization

The samples were tested at the central laboratory of the Geological Surveys of Ethiopia (GSE).

a. Kentry chemical analyses of gypsum: To determine the quality of the potential target, the samples were tested for CaO, SO_3 and H_2O and the following average values were obtained as in Table 15:

Table 15: Compositional analysis of Kentry gypsum

Compounds	CaO(%)	SO_3(%)	H_2O(%)	Others (%)	Total (%)
Compositions	37.40	49.98	11.03	1.59	100
Literature Review	32.6	46.5	20.9	-	100

This implies gypsum contains higher composition of SO_3 and lower composition of water. Therefore, these values are within acceptable limit to exploit the occurrence as a raw material for POP and chalk. Moreover, the average compositional analysis results of the crushed samples compositions were tested and the results are in Table 16.

Table 16: Compositional analysis of Kentry gypsum after crushed

Compounds	CaO(%)	SO_3(%)	H_2O(%)	LOI (CO_2) (%)	Others (%)	Total (%)
Compositions	38.47	44.18	7.96	6.52	2.87	100
Literature Review	32.6	46.5	20.9	-	-	100

As it can seen from the average values the water content tends to be lower and LOI tends to be relatively higher, which indicates that the crushed samples contain limestone fragments (impurities) that consequently increase in LOI and reduce in water content.

b. Filiklik (Blue Nile Gorge) chemical analyses of gypsum: The compositional analyses of this gypsum are the following:

Therefore, the gypsum occurrence is within acceptable limit to exploit as a raw material for POP and chalk.

c. Yogef chemical analyses of gypsum: The compositional analyses are described for both lower and upper gypsum:

Table 17: Compositional analysis of Filiklik gypsum

Compounds	CaO(%)	SO_3(%)	H_2O(%)	Others (%)	Total (%)
Compositions	38.23	50.43	10.62	0.72	100
Literature Review	32.6	46.5	20.9	-	100

Table 18: Compositional analysis of lower and upper Yogef gypsum

Compounds(%)	CaO	SO_3	H_2O	LOI	SiO_2	Fe_2O_3	AlO_3	Others	Total
Lower gypsum bed	30.75	44.57	16.87	5.27	0.92	0.12	0.11	1.39	100
Upper gypsum bed	30.99	45.61	18.67	3.70	0.79	0.09	0.11	0.04	100
Literature Review	32.6	46.5	20.9	-	-	-	-	-	100

Therefore, the gypsum occurrence is within acceptable limit to exploit as a raw material for POP and chalk. The comparisons of all the above tables in terms of their distances from Addis Ababa and the resources capacity were summarized in the Table 19 below. From this table the gypsum quarried from Kentry was selected in terms of distances from Addis Ababa and resources capacity. The Yogef (Upper gypsum bed) contains water content than the others whereas Filiklik gypsum contains more percentages of CaO and SO_3 than the rest.

Table 19: Comparison of compositional analyses for the raw materials

Description	Dist.from A.A	CaO(%)	SO_3(%)	H_2O(%)	Access	Resources(Tons)
Kentry	63km	37.40	49.98	11.03	Good	Up to 10^6
Filiklik	202km	38.23	50.43	10.62	Good	(3-5)*10^5
Yogef (LGB)	158km	30.75	44.57	16.87	Good	218,350
Yogef (UGB)	158km	30.99	45.61	18.67	Good	796,068
Literature Review	-	32.6	46.5	20.9	-	-

4.2 Statistical Results

Particle size (A), calcinations temperature(B) and moisture content(C) were chosen as the independent variables while the bulk density (Y_1) and breaking strength (Y_2) were selected as the response (dependent variable) in the present study. According to the CCD, a series of experiments was conducted for exploring different combination parameters and for evaluating the combined effects of these factors.

The aim of present work was to achieve optimized formulations for quality chalk by determining the effects of important factors and their interactions on physicochemical properties. Meanwhile the influence of different factors had been evaluated by making changes in these factors and three of the most significant factors had been chosen as the independent variables. The experiments were conducted and the results were collected according to the experimental design output of the software to minimize equipment and human error.

4.2.1 Optimization using Desirability

In this study, CCD was used for response surface optimization with three process variables (particle size, calcinations temperature and moisture content) at three levels. Designs using CCD are usually very efficient in terms of the number of required runs and therefore are less expensive to run compared to general factorial design.

In most RSM problems, the form of the relationship between the response and the independent variables is unknown. Thus, the first step in RSM is to find a suitable approximation for the true functional relationship between dependent and independent variables. Usually, a low-order polynomial in some region of the independent variables is employed. If the response is well modeled by a linear function of the independent variables then the approximating functions is the first order model. If there is curvature in the system, then a polynomial of higher degree must be used, such as the second-order model.

RSM is a sequential procedure. Our objective here is to lead our experiment rapidly and efficiently along a path of improvement toward the general vicinity of the optimum. Once the region of the optimum has been found, a more elaborate model, such as the second-order model may be employed, and an analysis may be performed to locate the optimum. The eventual objective of RSM is to determine the optimum operating conditions for the system or to determine a region of the factor space in which operating requirements are satisfied.

A 20 runs with three replicate and a total of 60 experiments were conducted for optimization of process parameters. The laboratory results of the experiment have been collected depending on the run order recommended by the Design-Expert software (version 7.0, Stat Ease) software and the average result of the three replicate was taken as one run and tabulated in the Table 20.

Analysis of variance was employed to determine the significance of the models. The test for the significance of the regression models, the test for significance on individual model coefficients, F-test and the lack-of-fit test were performed using the same statistical software package to obtain the best fit. The larger the value of F and the smaller the value of p, the more significant is the corresponding coefficient term [71]. The coefficient of determination (R^2) is defined as ratio of sum of squares due to regression to the total sum of squares and is interpreted as the proportion of the variability in the data explained by the ANOVA. The words lack of fit refers to the fact that the simple linear regression model may not adequately fit the data [72]. Prediction error sum of squares (PRESS) is a measure of how well the model for the experiment is likely to predict the responses in a new experiment. Smaller values of PRESS are desirable[73]. Verification of optimized param-

eters and predicted values were done in triplicate to confirm the validity of the models.

The results were analyzed by applying the coefficient of determination (R^2), analysis of variance (ANOVA) and response plots. The response surfaces help to understand the effect of process variables and parameters up on the responses and direction the response in which it increases or decreases. The actual experimental values were quantitatively compared with the predicted values to compute the prediction error (%). Linear correlation between the actual and predicted values of each response was evaluated from the error (%) to obtain the R^2 values for optimum model validation[68].

The optimized formulation was selected making use of desirability functions. Each

Table 20: Statistical results for actual and experimental

Run	**A:PS**	**B:CT**	**C:MC**	**Bulk Density(g/cm^3)**	**Breaking Strength (N/cm)**
1	45	140	6	1.206	13.3
2	70	140	6	1.150	14.0
3	95	135	7	1.104	12.0
4	70	135	6	1.166	13.8
5	70	145	7	1.125	11.8
6	70	145	6	1.135	14.3
7	95	145	7	1.064	12.6
8	70	140	7	1.145	11.5
9	45	145	5	1.193	15.9
10	70	145	5	1.140	16.6
11	95	135	5	1.120	17.0
12	95	145	5	1.084	17.5
13	95	140	6	1.093	14.9
14	70	140	5	1.154	16.4
15	45	145	7	1.181	10.8
16	45	140	5	1.210	15.7
17	95	140	7	1.088	12.4
18	95	145	6	1.075	15.2
19	45	135	7	1.214	10.2
20	45	135	5	1.225	15.5

response is associated with its own partial desirability function. If the value of the response is optimum, its desirability equals 1, and if it is totally unacceptable, its value is zero[69]. Thus the desirability for each response can be calculated at a given point in the experimental domain. The optimum is the point with the highest value for the desirability. Various 3-D response surface graphs and contour plots were studied over the entire experimental region, to select optimum checkpoint formulations for validation of the experimental domain and polynomial equation.

From over view of the results in Table 20, the maximum bulk density (1.225 g/cm^3) has been obtained at PS (45μm), CT (135^oC) and MC (5%) whereas the minimum bulk density (1.064 g/cm^3) has been observed at PS (95μm), CT (145^oC) and MC (7%). Similarly, the maximum breaking strength (17.50 N/cm) has been obtained at PS (95μm), CT (145^oC)

and MC (5%) whereas the minimum breaking strength (10.20 N/cm) has been observed at PS (45μm), CT (135^oC) and (MC 7%). All experiments were carried out in a randomized order to minimize the effect of unexpected variability in the observed response due to extraneous factors.

The numerical optimization of the software has been chosen in order to find the specific point that maximizes the desirability function. The desired goal was selected by adjusting the weight or importance that might alter the characteristics of a goal. The goal fields for responses have five options: none, maximum, minimum, target and within range and given Table 21.

Table 21: Optimization criteria used in this study

Name	**Unit**	**Goal**	**Lower Limit**	**Upper Limit**
Particle Size	(μm)	in range	45	95
Calcinations Temperature	^oC	minimize	135	145
Moisture content	%	in range	5	7
Bulk Density	g/cm^3	target	1.064	1.225
Breaking Strength	N/cm	target	10.2	17.5

Model selection for further analysis: Two different tests, namely, sequential model sum of squares and model summary statistic were performed to check the adequacy of the models generated from the obtained data. The result from the sequential model selects the highest order polynomial where the additional terms are significant and the model is not aliased. Model summary statistics output showed that the values for the R^2 and adjusted R^2 were the highest compared with the other models while the cubic model was disregarded as it is aliased [70]. The fitness of the models was determined by R^2 and adjusted R^2 such that the higher value of R^2 the better the model. Therefore, the two models suggested that 2FI for bulk density and quadratic model for breaking strength provided an excellent explanation for the relationship between the independent variables and the corresponding response. The results from these two models tests indicated that these models could be used to navigate the design space and provide a good description of the experimental data and for this reason, it was selected as the most appropriate model for further analysis.

4.2.2 Development of Regression Model Equation

The coefficients of 2FI and quadratic equations which included only statistically significant (p<0.05) coefficients for each response were generated as in Appendix B (b and c). The equation 5 and 6 were formed from that coefficient and a positive coefficient indicates that response is favored, while a negative value indicates an inverse relationship between the factor and the response.

The second order equations predicting the optimal point was achieved according to the

central composite experimental design and input variables, and the empirical relationship between the response and the independent variables in the coded units based on the experimental results was given below. Coded factors indicate when the minimum, mid and maximum values of the factors are represented by -1, 0 and +1, respectively instead of actual values. The output response surface bulk density and breaking strength are a function of the process parameters such as a particle (A), calcinations temperature (B) and moisture content (C).

I. Bulk Density

pper gypsum bed Density = Y_1 = [+1.15 - 0.056*A - 0.017*B + 7.000E-003*C - 1.377E-003 *A*B - 1.695E003*A*C] .....(5)

From equation (5) the coefficients of A and B were negative whereas the coefficient of C is positive. Therefore, particle size and calcinations temperature was negatively affect the bulk density whereas moisture content was positively affect bulk density. The coefficients of the two factors interaction also negative showing that they have negative effect on the response.

II. Breaking Strength

Breaking Strength = Y_2 = [+14.06 + 0.84*A + 0.26*B - 2.51*C + 0.071*A*C + 0.051*B*C - 0.12*C^2] .....(6)

From equation (6) the coefficients of A and B were positive, whereas the coefficient of C was negative. Therefore, breaking strength was positively affected by particle size and calcinations temperature, and negatively affected by moisture content. The coefficients of the two factors interaction (AC, BC) were positive and the quadratic C^2 was negative effect on the breaking strength.

Confirmation Experiments

The data were also analyzed to check the correlation between the experimental and predicted values for the responses as shown in Table 20. The experimental values were the measured response data for the runs designed by the Central composite model, while the predicted values were obtained by calculation from the 2FI and quadratic equation for bulk density and breaking strength respectively. The results show that experimental data and data obtained by regression equation closely correlate with each other, which validate the regression equations developed.

4.3 Data Analysis of Process Parameters

Data obtained from the experiment were analyzed with the help of software for optimization of processing and operating parameters with respect to the responses of the quality chalk. Multiple regressions analysis and ANOVA were conducted for fitting the model and to examine statistical significance of the model terms for each response. The statistical analysis of data and three-dimensional plotting were performed using the software. The adequacies of the models were determined using model analysis, lack of fit test, R^2,

Adjusted R^2, Precision R^2, Adjusted precision and F-test analysis as outlined.

4.3.1 Data Analysis for Bulk Density

From the ANOVA table 22, the data was tested by different values. The Model F-value (3107.95) with low probability value (p < 0.0001) statistically signifies the model. There is only a 0.01% chance that a "Model F-Value" this large could occur due to noise. The linear terms of particle size, calcinations temperature and moisture content, and the interaction between particle size and calcinations temperature; particle size and moisture content were significant at p < 0.0001 level and all the quadratic terms were not. Values of "Prob > F" less than 0.05 indicate model terms are significant. In this case A, B, C, AB and AC are significant model terms. Values greater than 0.10 indicate the model terms are not significant.

Table 22: Analysis of variance table for improved model for Bulk Density

Source	**Sum of squares**	**Df**	**Mean square**	**F-value**	**P-value**	**Remark**
Model	0.048	5	7.934×10^{-3}	3107.95	$<$0.0001	significant
A	0.039	1	0.039	12793.29	$<$0.0001	
B	3.622×10^{-3}	1	3.622×10^{-3}	1201.90	$<$0.0001	
C	6.707×10^{-4}	1	6.344×10^{-4}	210.52	$<$0.0001	
AB	1.674×10^{-5}	1	1.656×10^{-5}	5.54	0.0348	
AC	2.814×10^{-5}	1	2.269×10^{-5}	9.34	0.0090	
Residual	3.917×10^{-5}	14	2.621×10^{-6}			
Lack of fit	2.265×10^{-4}	2	1.887×10^{-5}	0.27	0.8907	not significant
Pure error	1.805×10^{-4}	4	1.805×10^{-4}			
Cor Total	48.6	19				

† $\mathbf{R^2}$ = 0.9991; Pred.$\mathbf{R^2}$ = 0.9980; Adj.$\mathbf{R^2}$ = 0.9988; and Adeq.Precision = 166.039

If there are many insignificant model terms (not counting those required to support hierarchy), model reduction may improve the model. An insignificant lack of fit 0.27 implies that the model was robust enough to explain the effects of the three parameters on bulk density. Model fitting with the help of Design-Expert software suggested that a two-factor interactions model provided the best fit, and the model was found to have insignificant lack of fit. This was desirable as we want a model that fit.
By selecting the step-wise regression method, which eliminates the insignificant model terms automatically, Tables 26 also show the other adequacy measures R^2, adjusted R^2 and predicted R^2. The fit of the model was checked by the coefficient of determination R2 for bulk density was calculated 0.9991, which is very close to 1, indicating that 99.91% of variability in the response could be explained by the model and only 0.09% were not explained by the model. CV (Coefficient of Variation) value to check the reliability and precision of the model obtained from the model was 0.15%.

In this case, the values of PRESS are considerably smaller. The Predicted R^2 of 0.9980 is in reasonable agreement with the Adjusted R^2 of 0.9988 with the difference of 0.08% that signifies a good adjustment of the theoretical values to the experimental data by the

model. Adequate Precision measures the signal to noise ratio and a ratio greater than 4 is desirable. The ratio of 166.039 was much greater than 4 that indicates an adequate signal and the model can be used to navigate the design space.

4.3.2 Data Analysis for Breaking Strength

The same procedure has been applied on this response as that of bulk density. By selecting the backward elimination procedure to automatically reduce the terms that were not significant, the resulting ANOVA table for the reduced quadratic model for breaking strength was displayed. The adequacy of the developed models was tested at 95 % confidence interval and the results of the linear and quadratic order response surface model fitting in the form of ANOVA was given in Table 23. The ANOVA results (Table 23) indicated that the model F-value of 4868.10 was greater than $F_{0.05}$ = 2.52, implying that most of the variation in the response could be explained by the regression equation and that the model was significant. Furthermore, the probability p<0.0001 also suggested that the model was 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 0.05 indicate model terms are also significant. The "Lack of Fit F-value" of 0.36 implies the Lack of Fit is not significant relative to the pure error. Non-significant lack of fit is good since we want the model to fit. In this case A, B, C, AC, BC and C^2 are significant model terms. Breaking strength was significantly affected by linear terms of the parameters at (p < 0.0001); the interaction between particle size and moisture content, calcinations temperature and moisture content, and the quadratic of the moisture content with effect of (p<0.005). The lower value of CV 0.39% indicates greater reliability of the experiments performed with smaller values of PRESS of 0.096.

Table 23: Analysis of variance table for improved model for Breaking Strength

Source	**Sum of squares**	**Df**	**Mean square**	**F-value**	**P-value**	**Remark**
Model	89.54	6	14.92	4868.10	$<$0.0001	significant
A	8.75	1	8.75	2853.46	$<$0.0001	
B	0.86	1	0.86	279.452	$<$0.0001	
C	83.41	1	83.41	27207.08	$<$0.0001	
AC	0.049	1	0.049	15.94	0.0062	
BC	0.025	1	0.025	8.27	0.0203	
C^2	0.061	1	0.061	19.87	0.0026	
Residual	0.040	13	3.066×10^{-3}			
Lack of fit	0.59	11	0.045	0.36	0.8799	not significant
Pure error	0.13	5	0.13			
Cor Total	89.58	19				

† $\mathbf{R^2}$ = 0.9996; Pred.$\mathbf{R^2}$ = 0.9989; Adj.$\mathbf{R^2}$ = 0.9993; and Adeq.Precision = 220.081

High multiple correlation coefficient (R^2) value of 0.9996, which is very close to 1, signifies a good model explanation where only 0.04% of total variation was not explained by the model. A reasonable agreement between the Predicted R^2 (0.9989) and Adjusted R^2 (0.9993) with 0.04% difference indicates a good correlation between the observed and

predicted values. Adequate Precision measures the signal-to-noise ratio, with a ratio greater than 4 considered as desirable. The Adequate Precision ratio of 220.081 obtained in this study indicates an adequate signal and this model can be used to navigate the design space. These insignificant model terms (not counting those required to support hierarchy) should be removed from the model itself and make it into an improved model.

4.3.3 Diagnostic test

Usually, it is necessary to check the fitted model to ensure that it provide an adequate approximation to the real system. Unless the model showed an adequate fit, proceedings with the investigation and optimization of the fitted response surface likely give poor or misleading results. The residuals from the least squares fit played an important role in judging model adequacy. The quality of the model developed could be evaluated from their coefficients of correlation. By constructing a normal probability plot of the residuals, a check was made for the normality assumption, was given (Fig. 10). All diagnostic

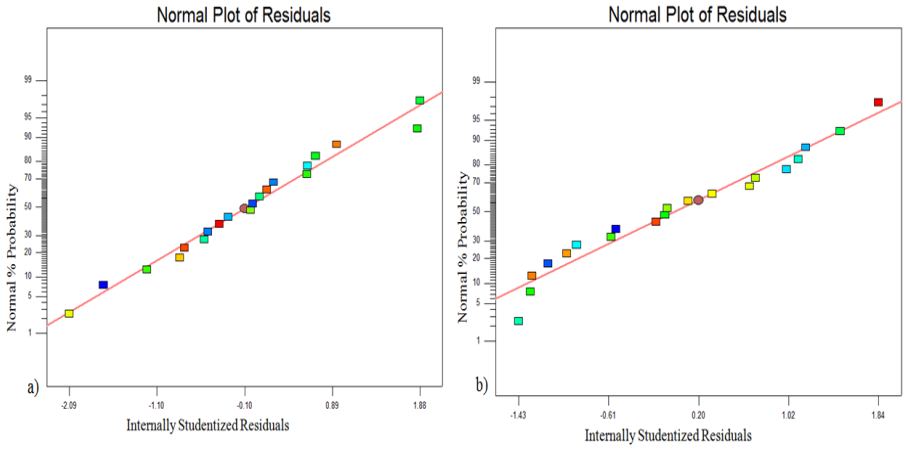


Figure 9: Plots of Residuals for a) bulk density b) breaking strength

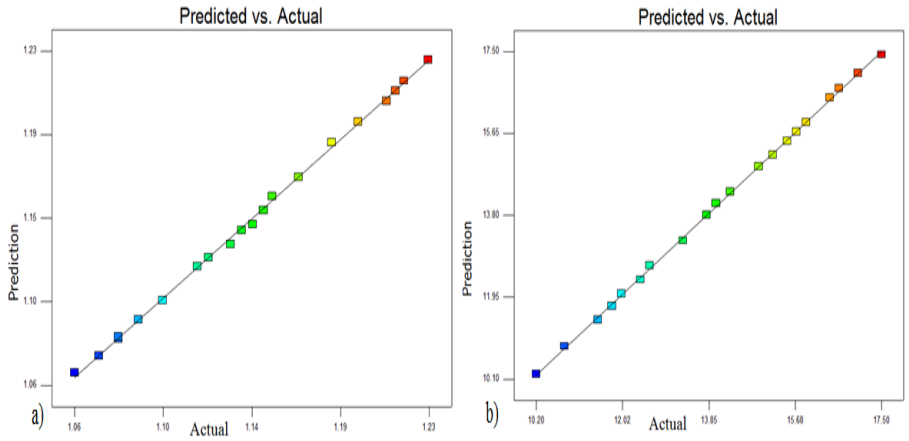


Figure 10: Plot of the actual and predicted values for a) bulk density b) breaking strength

plots were also tested for both responses for adequacy of the models (normal plot of residuals, residuals versus predicted value, residuals versus factor, box Cox plot, studentized residuals, leverage, etc.). As both models showed a satisfactory fit, normal proba-

bility plot of the residuals were generated to check the normality of the residuals. The residuals were studentized and values which were greater than +2 and less than -2 were considered as large. As both models showed a satisfactory fit, normal probability plot of the residuals were generated to check the normality of the residuals (Figure 9). Obtaining a smaller residual value is preferred as this shows the degree of deviancy from predicted model. It is clear from figure that the residuals followed normal distribution well as majority of the data points followed the fitted line closely with no reasonable outliers.

The actual and predicted values for both responses were given in Figure 10. From the figures the predicted data of the response from the empirical model is in good agreement with the observed ones in the range of the operating variables. It shows how precisely bulk density and breaking strength were modeled because the entire points lineup nicely. A line of unit slope, i.e. the line of perfect fit with points corresponding to zero error between predicted and actual values. This plot therefore visualizes the performance of the correlation in an obvious way[60].

Correlation Matrix for Optimization

The correlation matrix is one of the most common and useful statistics. The correlation coefficient measures the strength of linear relationships between the variables [72]. The correlation coefficient is always between -1 and +1. The closer the correlation to +/-1, the closer to perfect linear relationships[67]. From Appendix B (d) almost all, the factor-to-factor correlation is zero. For the factor -to-factor correlation of diagonal values close zero are better which is satisfied in this case.

4.3.4 Effect of Processes Parameters on the Responses

The detailed main and interaction effects for both the responses are discussed in the following sections. It should be noted that if a particular parameter does not influence the responses during the course of evaluation, it gets eliminated.

Perturbation Plot

Perturbation plot shows how a function of a certain factor responded as the level of that factor changes, when the other factors are fixed at their optimum levels. A steep slope or curvature in the plots indicates the sensitivity of the response factor. Perturbation plot for bulk density and breaking strength (Appendix C (a)) are used to assess the effect of each factor on the responses. For the bulk density, response increment was observed as particle size and calcinations temperature were decreased; but moisture content plays minimal influence on the response. For breaking strength, by comparing the slope of every factor, the increment of the response was observed as particle size and calcinations temperature factors were increased, and the moisture content decreased.

Influence of Individual Effect

The individual effect of particle size, calcinations temperature and moisture content towards bulk density and breaking strength were plotted in the Appendix B (b and c) respectively. The bulk density was decreased from 1.195g/cm^3 to 1.101g/$cm^3$with the strictly increasing particle size from 45μm to 95μm. It also decreased from 1.165g/cm^3 to 1.125g/cm^3 with smoothly increasing of calcinations temperature from 135^oC to 145^oC and decreasing of moisture content from 7% to 5%. This is due to the negative coefficient of two-factor interaction model as shown in equation 5. It also indicates that the experimental value must consider running effect of particle size and calcinations temperature at a higher level to minimize the bulk density. At the same time, maximum bulk density was found from the lower value of particle size and calcinations temperature and higher value of moisture content. Similarly, breaking strength was increased from 13N/cm to 15N/cm with increasing of particle size from 45μm to 95μm and 13.8N/cm to 14N/cm with smoothly increasing of calcinations temperature from 135^oC to 145^oC and it was decreased from 16N/cm to 11N/cm with strictly increasing moisture content from 5% to 7%. This due to the positive coefficient of two factor interactions and negative coefficient of quadratic moisture content in the equation 6. Therefore, the maximum breaking strength was obtained at higher value of particle size and calcinations temperature, and lower value of moisture content.

Influence of Interaction Effect

The interaction effects of variables on responses were studied by plotting two factor interactions (Appendix C(d and e)), Contour plot (Appendix C (f and g)) and 3-D surface curves (Figures 12 and 14) against any two independent variables and keeping another variable at its central (0) level. The 3-D curves of the calculated responses plots from the interactions between the variables are shown in Figure 12(a - c), using the statistical software. The corresponding contour plots (Appendix C (f and g)), represented by the projection of the response surfaces in the x-y plane, provide a straightforward determination of the effects of the independent variables on the dependent variable.

I. Bulk density

Interaction between process variables can affect the bulk density and these effects have been seen statistically and graphically. a. Effect of Particle Size - Calcinations Temperature (AB) on Bulk Density

AB = 2/4[1.10 + 1.23 + 1.07 + 1.19 - 1.18 - 1.08 - 1.21 - 1.12] = 0.0012

b. Particle Size - Moisture Content (AC) Effect

AC = 2/4[1.23 + 1.21 + 1.08 + 1.07 - 1.18 - 1.19 - 1.10 - 1.12] = 0.0024

c. Calcinations Temperature - Moisture Content (BC) Effect

BC = 2/4[1.23 + 1.12 + 1.18 + 1.07 - 1.19 - 1.08 - 1.21 - 1.10] = 0

These results show that the interactions of particle size - calcinations temperature and particle size-moisture content have minimum effect on bulk density whereas the inter-

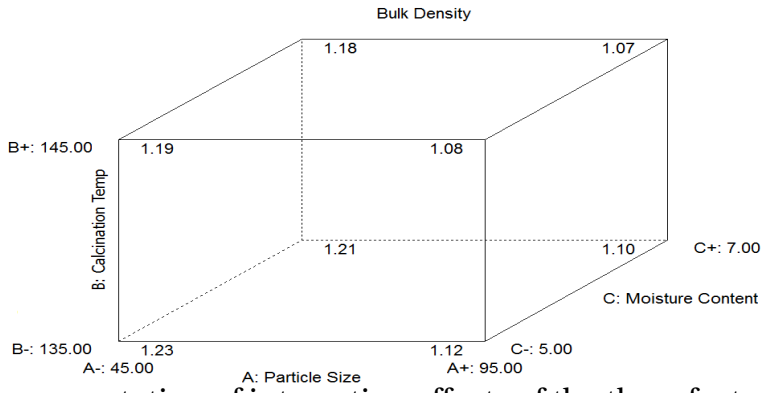


Figure 11: Cubic representation of interaction effects of the three factors on bulk density

action of calcinations temperature - moisture content has not. This was shown graphically in Appendix C (d). From the graph, it was seen that the lines are almost parallel that shows there was no significant interaction effect between the two factors.

Response Surface for Bulk Density

Figure 12(a-c) shows the interactions effects of particle size, calcinations temperature and moisture content on the response variable of bulk density. The response surface plots in three figures were both part of a diagonal, exhibiting a minimum and maximum ridge, respectively, in the investigated domain. In each response surface, the optimum values of variables could be analyzed by the saddle point or by determining the maxima formed by the x- and y-coordinates.

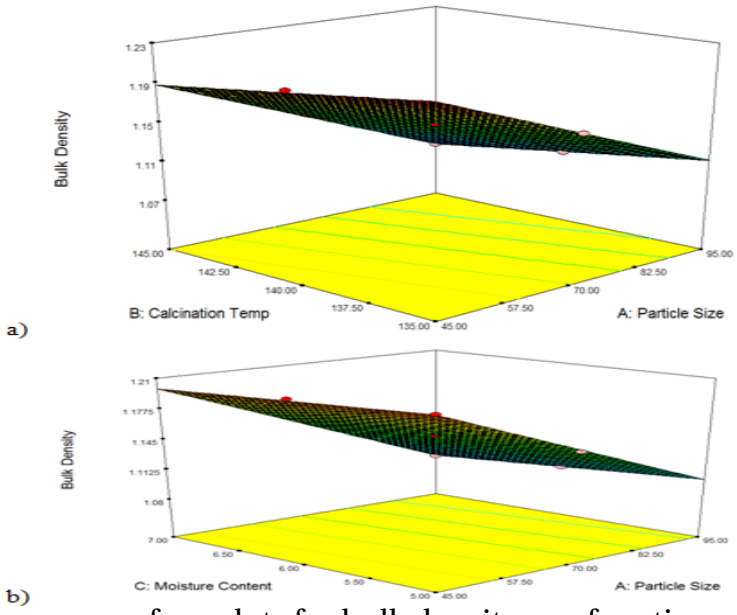


Figure 12: Response surface plots for bulk density as a function of three factors

Figure 12a depicts the interaction effect of particle size and calcinations temperature on bulk density at constant moisture content of 6%. It has been seen that, at given constant moisture content, bulk density uptake increased with decreasing calcinations temperature smoothly and particle size strictly. However, increasing calcinations temperature and particle size decreases bulk density.

Figure 12b depicts the interaction effect of particle size and moisture content on bulk density at constant calcinations temperature of 140^oC. When size of the particle decreased and the moisture content increased, the bulk density was increased. However, increasing particle size and decreasing moisture content decreases bulk density.

Figure 12c depicts the interaction effect of calcinations temperature and moisture content on bulk density at constant particle size of 70μm. As calcinations temperature increased and moisture content decreased, the bulk density was decreased. However, decreasing calcinations temperature and increasing moisture content increase bulk density.

Generally, it was understood from these three figures that when the size of the particle is small, minimum temperature was needed to evaporate moisture content. As maximum percentage of moisture content was evaporated from the sample, the product become dry. When the size of the particle is large, it needs maximum temperature to evaporate the moisture content it has. As maximum percentage of moisture content was left in the particle, the product become humid. This is the reason why it was wanted to take the optimum values of these variables from the range to get the targeted bulk density.

II. Breaking strength

Interaction between process variables can affect the breaking strength and these effects have been seen statistically and graphically.

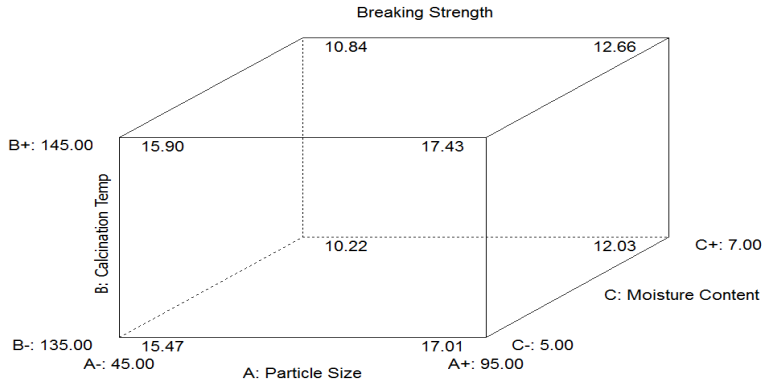


Figure 13: Cubic representation of interaction effects of the three factors on breaking strength

a. Particle Size - Calcinations Temperature (AB) Effect

AB = 2/[15.47 + 12.03 + 12.66 + 15.90 - 10.84 - 17.43 - 10.22 - 17.01] = 0

b. Particle Size - Moisture Content (AC) Effect

AC = 2/4[15.47 + 10.22 + 17.43 + 12.66 - 17.01 - 12.03 - 15.90 - 10.84] = 0.007

c. Calcinations Temperature - Moisture Content (BC) Effect

BC = 2/4[15.47 + 17.01+ 10.84 + 12.66 - 10.22 - 12.03 - 15.90 - 17.43] = 0.05

These results show that the interactions of particle size - calcinations temperature has no

effect on the breaking strength whereas the interactions particle size - moisture content and calcinations temperature - moisture content have minimum effects on the response. This is shown graphically in Appendix C (e). From the graph, it was has been seen that the lines are almost parallel that shows there is no significant interaction effect between the two factors.

Response Surface for Breaking Strength

Figure 14(a-c) show interaction effects of particle size, calcinations temperature and moisture content on the response variable of breaking strength. The response surface plots in figure 14a was part of rectangle and that of figures 14b and 14c were part of a diagonal exhibiting a minimum and maximum in the investigated domain. In each response surface, the saddle could analyze the optimum values of variables.

Figure 14a depicts the interaction effect of particle size and calcinations temperature on

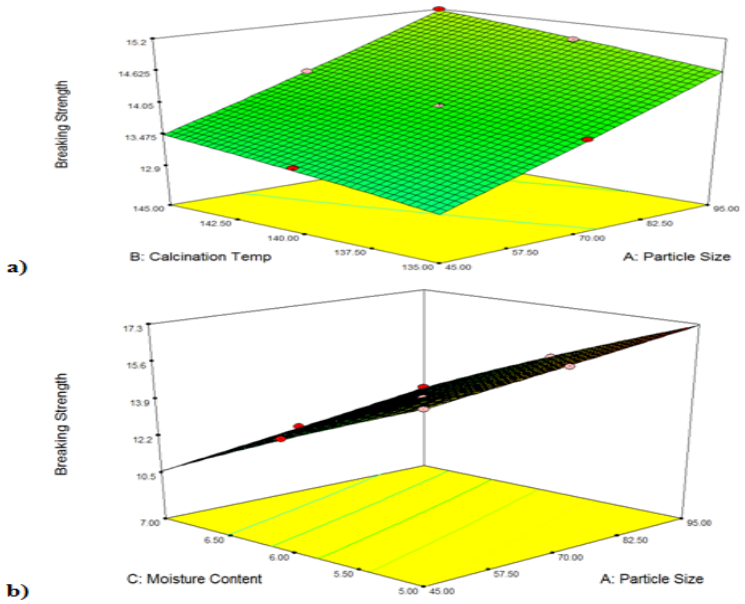


Figure 14: Response surface plots for breaking strength as a function of the three factors

breaking strength at constant moisture content of 6%. As particle size and calcinations temperature increased, breaking strength also increased. However, decreasing calcinations temperature and particle size decrease breaking strength.

Figure 14b depicts the interaction effect of particle size and moisture content on breaking strength at constant calcinations temperature of 140^oC. As the size of the particle increased and moisture content decreased, the breaking strength was increased. However, decreasing particle size and increasing moisture content decrease breaking strength.

Figure 14c depicts the interaction effect of calcinations temperature and moisture content on breaking strength at constant of particle size 70μm. When calcinations temperature increased and moisture content decreased, the breaking strength also increased.

However, decreasing calcinations temperature and increasing moisture content decrease breaking strength.

It was understood from these figures that; when there is much moisture content in the sample, the product become soft and it can be broken easily. But as the moisture content was minimized in the sample, the product become hard, and the chalk has a sound of disturbance when use with it.

Similarly, as the size of the particle was reduced to very small diameter, the product can break easily. But when the diameter was increased, the final product may not break as such. This is the reason why it was wanted to take the optimum values of these variables from the range to get the targeted breaking strength.

4.3.5 Optimization of the Process Parameters

The primary objective of the present study was to find the optimum process parameters to get optimum responses of good quality chalk. Numerical optimization step in CCD was used to predict the optimum values of bulk density and breaking strength by the adjusted model. The goals for variables of particle size and moisture content were set as "in range" whereas calcinations temperature was "minimize". The goals for the responses (bulk density and breaking strength) were set as "target" because the optimum values of bulk density and breaking strength were neither at maximum point nor at minimum point. Desirability function was used to identify the optimum levels of factors and to get maximum desirable response. The individual desirability and the combined desirability of both responses were reported in table below via numerical optimization.

Table 24: Optimum solutions obtained depending on the desirability

S.N	Particle Size	Calcination Temperature	Moisture Content	Bulk Density	Breaking Strength	Desirability
1	85.55	137.00	6.25	1.1445	13.85	0.998
2	80.35	139.55	6.11	1.1442	13.85	0.992
3	80.35	142.64	6.11	1.1452	13.85	0.948
4	80.62	139.25	5.98	1.1441	14.25	0.925
5	80.81	138.06	6.57	1.431	13.04	0.915

The standard ranges for bulk density and breaking strength are between 1.112 - 1.201 g/cm^3 and 10 - 20N/cm respectively[74][75]. The values, which are out of these ranges, are not considered in the optimization domain. The values that are in these ranges are identified by their desirability, the value that has maximum desirability was taken as an optimum value. Therefore, as it can be seen from Table 24 below, a number of solutions was produced by the software and ranked according to their desirability. The optimized

batch was selected with maximum combined desirability value i.e. 0.998 using the software.

The solutions with the highest desirability were chosen and the optimum values were 1.1445g/cm^3 and 13.85N/cm for bulk density and breaking strength respectively at 85.55μm particle size, 137^oC calcinations temperature and 6.25% moisture content as optimum value of process parameters. The experimental values of the response variables obtained from the optimized formulations were close and in linear with the predicted values.

4.3.6 Validation of Optimum Results

An experiment was set up to validate the optimized condition. As shown in Table 24, the experimental data were in good agreement with the predicted values for bulk density and breaking strength. Relative error between predicted and experimental values fell at 0.39% and 1.21% for bulk density and breaking strength respectively. The verification value for responses obtained is within 99% of predicted values which clearly showed that the model fitted the experimental data very well and therefore optimized efficiently within the specified range of process parameters. The validation results demonstrated that the models developed are quite accurate as the percentages of error in prediction were in a good agreement. Thus, the low magnitude of error in this study improves the significant analytical ability of the model.

Table 25: Validation test results

PS(μm)	**CT(^oC)**	**MC(%)**		**BD(g/cm^3)**	**BS(N/cm)**
85.55	137	6.25	Predicted	1.1445	13.85
			Experimental Validated	1.1418	14.02
			Error %	0.39	1.21

Once it was understood that the optimum process parameters were obtained from the experimental result, it was compared with practical chalk factory in Ethiopia as in Table 26. From this table there was a difference between these results and that of the practical one. This is the result of this thesis, which found the optimum parameters that improve the quality of the existing one. Therefore, the quality of chalk produced from the optimum parameters was strictly different from existing. The comparison of the optimum values of the present thesis with the values of literature review was tabulated in Table 26.

Table 26: The comparison of this result with the literature review

	PS(μm)	**CT(^oC)**	**MC(%)**	**BD(g/cm^3)**	**BS(N/cm)**
This thesis result	85.55	137	6.25	1.1445	13.85
Literature review	90	135	7	1.14	14.02

As a result of this paper, the size of particle was reduced from 90μm to 85.5μm, calcinations temperature was increased from 135^oC to 137^oC and the moisture content was

reduced from 7% to 6.25%. Because of changing these parameters, the responses also changed as breaking strength of the chalk decreased from 14N/cm to 13.85N/cm and the bulk density was increased from 1.14g/cm^3 to 1.1445g/cm^3.

Changing these responses eliminated the emits a shrill, piercing and scratching sound when applied on the blackboard. It also improved the problem of the dryness and difficulties of using chalk. It eliminated the fragmentizes and turns to dust or powder. It was easily wipe off from the blackboard and broken pieces were not found in the package.

5 FEASIBILITY STUDY OF IMPROVED QUALITY CHALK

5.1 Product Description

Chalk sticks are used in all schools and colleges everywhere. It has an indispensable role in the teaching and learning process being the medium through which the knowledge of the teacher is transferred to the students. It is a substance used for drawing and writing on rough surfaces as it readily crumbles leaving particles that stick loosely to these surfaces. Chalk also called blackboard chalk often supplied in sticks of about 80mm long is made from calcium sulphate dihydrate [44]. Ethiopia has nine region with two federal city totally eleven regions. But there is no chalk industry in any region except Addis Ababa. The other ten regions have transported chalk sticks from Addis Ababa. Chalk sticks are made mainly from gypsum, which is found in the many of the regions, and there is every economic reason for producing chalk sticks [45].

The demand for this product is expected to increase because there is high expansion of educational institution throughout the country. The consumption of chalk sticks will increase as the number of students and schools increase. The main raw material for chalk sticks is plaster of Paris or calcium hemi-hydrate, which is obtained by calcinating gypsum. Gypsum is found in many parts of the region in abundant quantity.

Demand Projection: The future demand for chalk depends on a number of interrelated factors.

Table 27: Demand and supply of chalk in Ethiopia (2000 - 2013)

Year	Demand (1000kg)	Supply (1000kg)
2000	375.8	278.3
2001	412.6	298.0
2002	460.2	377.7
2003	473.5	380.4
2004	490.0	392.0
2005	514.5	409.5
2006	540.4	411.6
2007	567.0	413.7
2008	595.7	415.1
2009	637.0	420.0
2010	681.8	424.2
2011	750.4	435.4
2012	825.3	451.5
2013	907.9	466.2

Source: FRDE Ministry of Education

These factors that are critical in determining the volume and trend of demand for chalk include:

- The overall economic development level and growth trend of the country;
- The performance and expansion of the education sector;
- Education budget and expenditure;
- Government policies on education; and
- Size of the population in general and the school age population in particular.

As it can be seen from Table 27, the demand of chalk and the supply wenclusion re far apart in terms of quantity. As the number of students increased from year to year rapidly in the country, the demand of chalk is increasing but the supply of chalk was not increasing with the demand.

Pricing: Currently, chalk factories in Ethiopia and the imported ones sell chalk for almost the same price, 8 Birr/gross for white chalk and 12 Birr/gross for colored chalk. Similarly locally produced gypsum sells up to 42.00 Birr per 25kg and imported gypsum fetches as much as 75 Birr per 25kg.

Plant Capacity: According to the market study and the economic scale of chalk manufacturing, the rated capacity of the plant is proposed to be 3200 gross per day of chalk of which 60% is white and 40 is colored chalk. The selected production capacity is based on 300 working days per annum and 16 hours per day by two shifts. The rest of calendar days are left for cleaning and maintenance

Production Program: The production program is based on the time required for the adjustment of feedstock, labor and equipment to the technology selected. The plant has planned to produce 75% of plant capacity during the first year; 85% of plant capacity during the second year, 100% of plant capacity during the third year and then after.

5.2 Quality Control

Quality is the rated ability of the product to perform its functions. It is the summary term for the products durability, reliability, precision, ease of operation and repair and other values of attributes. Quality assurance is an important and necessary function in any manufacturing company. It provides the necessary know-how to the plants so they can deliver finished products within the highest standards. The quality control department in a plant provides important services by maintaining a high standard of product quality and by giving proper training to the plant personnel. Today's market is highly

competitive. Any company is likely to lose business if it does not possess the capability of supplying customers with high-quality product at a competitive price. Therefore, more than ever before, it is important that the personnel in chalk production plant be well informed of the techniques of parameter processing, manufacturing, and quality and cost management [55].

To produce finished products with zero defects, the incoming raw materials must meet all specifications. Poor quality of gypsum prevents production of the best-quality finished product at a low cost and increases refining losses. Process operating standards are critical in order to obtain improved chalk quality with the proper functional properties and stability.

5.3 Material and Energy Balance

Material and energy balances are the basis of process design. A material balance taken over the complete process will determine the quantities of raw materials required and products produced. Balances over individual process units set the process stream flows and compositions. They are also useful tools for the study of plant operation and trouble shooting. They can be used to check performance against design; to extend the often-limited data available from the plant instrumentation; to check instrument calibrations; and to locate sources of material loss. On the other hand, in process design, energy balances are made to determine the energy requirements of the process: the heating, cooling and power required. In plant operation, an energy balance (energy audit) on the plant will show the pattern of energy usage and suggest areas for conservation and savings [73].

In the case of this research, the need to conduct material and energy balances on major unit operations that scale up all the parameters used in the laboratory resulted in the annual production of the plant; in order to design the size of the equipment that helped in estimating purchased equipment cost. In addition to that, they helped in calculating the material, auxiliary and utility costs. Generally, they are needed to estimate economic analysis; profitability and financial feasibility of the processing plant.

5.3.1 Material Balance

Material balance calculations are based on the principles of conservation of mass that states that matter can be neither created nor destroyed but may go under physical or chemical change. Material balance can be done on the size reduction process, calcinations, mixer, moulding and drying. General Material balance assumptions:

- 2 shifts per day
- 8 working hours per shift

- 16 working hours per day
- 300 working days per year
- 3,200 gross per day or 2,240 kg/day of capacity
- 3:2 percent ratio of white to colored chalk

The material balance was done by starting from the product gained and gone backward to determine the amount of raw materials required for processing the plant daily assuming the production capacity 2,240 kg/day.

1. Dryer: Assuming 58% of the total feed is evaporated in dryer.
Wet chalk = vapor + dry chalk

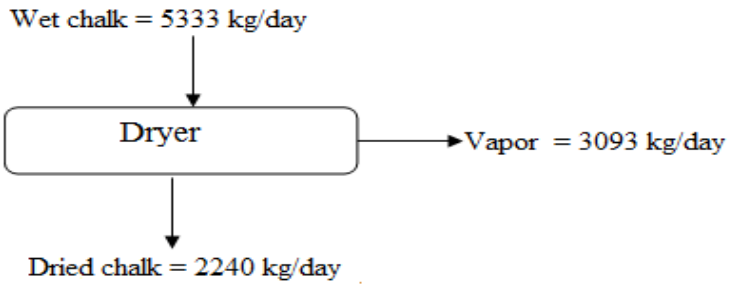


Figure 15: Material balance for dryer

but Vapor = 0.58 * wet chalk
Wet chalk = 0.58 * Wet Chalk + Dry chalk
=> 0.42 Wet chalk = Dry chalk = 2240 kg/day
Wet chalk = 5,333 kg/day and
Vapor = 0.58 * 5333kg/day = 3,093 kg/day

2. Moulding: there is 1% loss in molding due to breakage.
POP Slurry + Wasted Chalk = Wet chalk

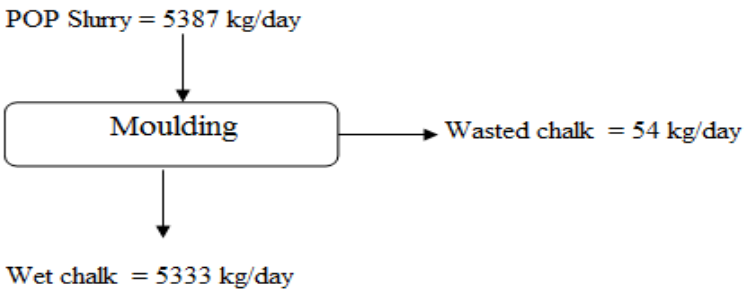


Figure 16: Material balance for moulder

but Wasted chalk = 0.01 * POP Slurry
POP Slurry = 0.01 * POP Slurry + Wet chalk
0.99 POP Slurry = Wet chalk = 5,333 kg/day
POP Slurry = 5387 kg/day and

Wasted chalk = 0.01 * POP Slurry = 54 kg/day

3. Mixer: In the laboratory result, the amount of clean water to POP is 6/5 in mixer and loss 0.2%.

POP Powder + Clean water = POP slurry + Wasted POP

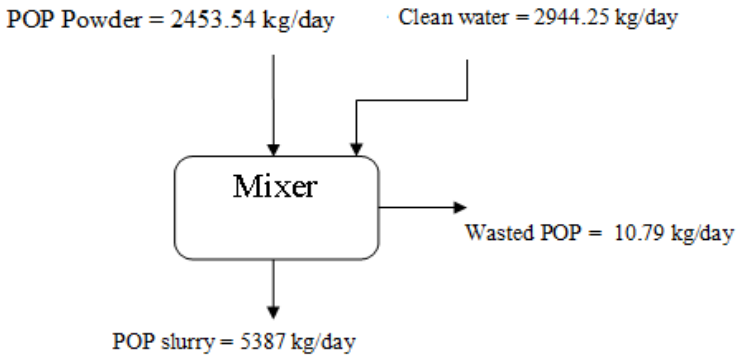


Figure 17: Material balance for mixer

But the Clean water/POP Powder = 6/5 = 1.2
=> Clean water = 1.2 * POP Powder and
Wasted POP = 0.002 * (POP Powder + Clean water) = 0.0044 * POP Powder
POP Powder + 1.2 * POP Powder = POP slurry + 0.0044*POP Powder
2.1956 POP Powder = POP slurry = 5,387 kg/day
POP Powder = 2453.54 kg/day and
Clean water = 1.2* 2,453.54 kg/day = 2,944.25 kg/day
Wasted POP = 0.0044 *2,453.54 kg/day = 10.79 kg/day

4. Pulverizing: Considering process loss of 1.1% during pulverizing,

POP = Loss + POP Powder but

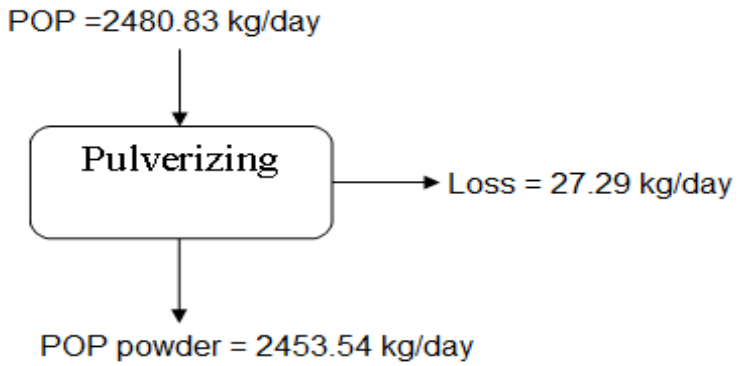


Figure 18: Material balance for Pulverizer

Loss = 0.011 * POP
POP = Loss + POP Powder
POP = 0.011 * POP + POP Powder
0.989 POP = POP Powder = 2,453.54 kg/day
POP = 2,480.83 kg/day and Loss = 0.011 * 2,480.83 kg/day = 27.29 kg/day

5. Calcinations/Kettle Calciner: According to laboratory result, 18% is losing during calcinations.

Gypsum powder = Water vapor + POP but

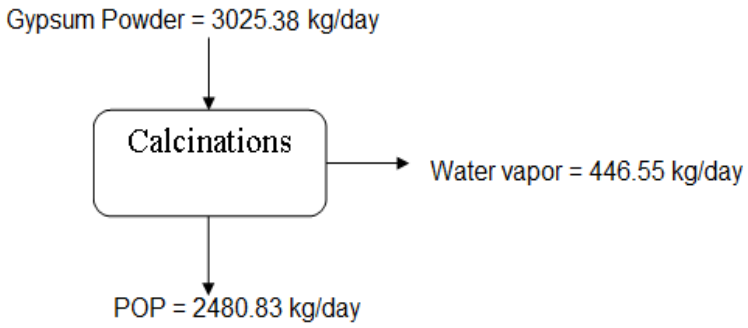


Figure 19: Material balance for kettle

Water vapor = 0.18 * Gypsum powder
Gypsum powder = Water vapor + POP
Gypsum powder = 0.18 * Gypsum powder + POP
0.82 Gypsum powder = POP = 2,480.83 kg/day
Gypsum powder = 3025.4 kg/day and Water vapor = 0.18*2,480.83= 446.55 kg/day

6. Grinding Mill: According to laboratory, result 1.5% loss in the grinding mill.

Gypsum from secondary crusher = Loss + Gypsum powder

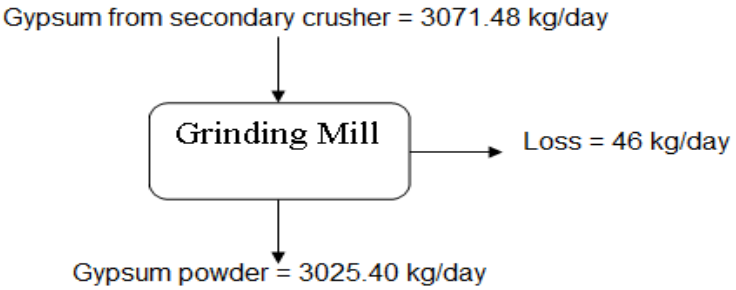


Figure 20: Material balance for grinding mill

But Loss = 0.015* Gypsum from secondary crusher
Gypsum powder from secondary crusher = 0.015* Gypsum from secondary crusher + Gypsum powder
0.985Gypsum from secondary crusher = Gypsum powder = 3,025.4 kg/day
Gypsum from secondary crusher = 3,071.48 kg/day and
Loss = 0.015* 3,071.47 = 46 kg/day

7. Secondary Crusher: the process loss of 0.55% during secondary crushing.

Gypsum from jaw crusher = Loss + Gypsum from secondary crusher
But Loss = 0.015* Gypsum from jaw crusher

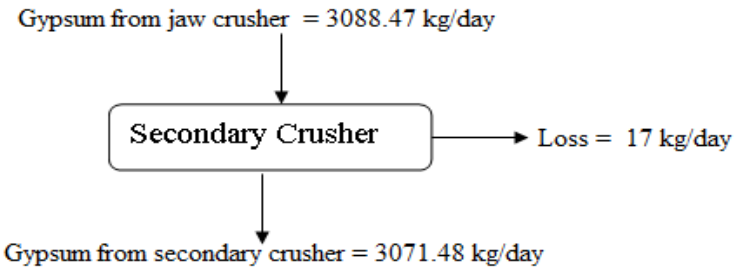


Figure 21: Material balance for secondary crusher

Gypsum powder from jaw crusher = 0.0055* Gypsum from jaw crusher + Gypsum from secondary crusher
0.9945 Gypsum from jaw crusher = Gypsum from secondary crusher = 3,071.48 kg/day
Gypsum from jaw crusher = 3,088.47 kg/day and
Loss = 0.0055* 3,071.47 = 17 kg/day

8. Jaw Crusher: According to laboratory result, 0.1% of the raw materials are losing during crushing and conveyance to the smaller crushing.
Gypsum rock = Gypsum powder from jaw crusher + Loss

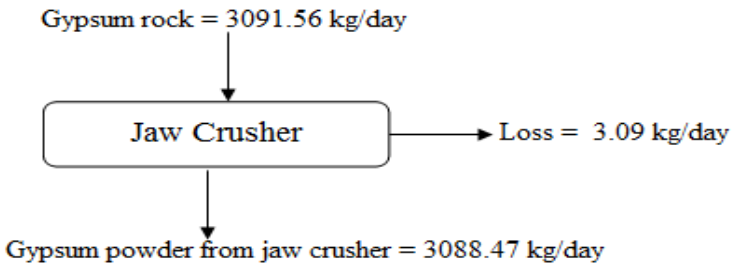


Figure 22: Material balance for jaw crusher

But Loss = 0.001* Gypsum rock
Gypsum rock = Gypsum powder from jaw crusher + 0.001* Gypsum rock
0.999 Gypsum rock = Gypsum powder from jaw crusher = 3,088.47 kg/day
Gypsum rock = 3,091.56 kg/day and
Loss = 0.001* 3,091.56 kg/day = 3.09 kg/day
Therefore, the plant needs the 3,091.56 kg raw materials in a day.

5.3.2 Energy Balance

The general equation for energy balance can be given as:
Accumulation = Energy out - Energy in + Generation - Consumption
Generally, to determine the cooling or heating requirements of any processing industry simplified equation can be met by ignoring the potential and kinetic energy of the system and considering the inlet and out let enthalpy of the system, heat added or removed from the system. All calculated energy values are the quantity of energy required to run one batch process[46].

Table 30. Thermodynamic properties of compounds

Energy balance can be done on the unit operation: calcinations unit and drying unit.

a. Energy Balance on Kettle Calciner

λ: Latent heat of evaporation = 2,256 kJ/kg

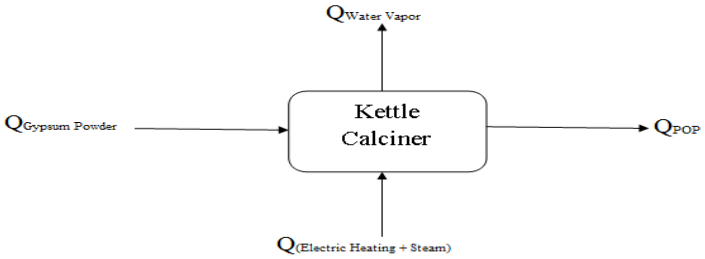


Figure 23: Energy Balance on Kettle Calciner

$Q_{GypsumPowder}$ + $Q_{(ElectricHeating+Steam)}$ = $Q_{WaterVapor}$ + Q_{POP}

$Q_{GypsumPowder}$ = $M_{GypsumPowder}$ $Cp_{,GypsumPowder}$ ($T_{GypsumPowder}$ - T_ref)

$Q_{GypsumPowder}$ = 3025.38 kg/day * 1.09 kJ/kg.K * (373.15K - 298.15K)

$Q_{GypsumPowder}$ = 2.473 X 105 kJ/day

Q_{POP} = M_POP $Cp_{,POP}$ (T_{POP} - T_{ref})

Q_{POP} = 2480.83 kg/day * 1.09 kJ/kg.K * (410.65 K - 298.15K)

Q_{POP} = 3.042 X 105 kJ/day

$Q_{WaterVapor}$ = MWv* $\lambda_{WaterVapor}$

$Q_{WaterVapor}$ = 446.55 kg/day * 2,256 kJ/kg

$Q_{WaterVapor}$ = 10.074 X 105 kJ/day

$Q_{GypsumPowder}$ + $Q_{(ElectricHeating+Steam)}$ = $Q_{WaterVapor}$ + Q_{POP}

$Q_{(ElectricHeating+Steam)}$ = $Q_{WaterVapor}$ + Q_{POP} - $Q_{GypsumPowder}$

$Q_{(ElectricHeating+Steam)}$= 1,007,416.8 kJ/day + 304,211.78 kJ/day - 247,324.82 kJ/day

$Q_{(ElectricHeating+Steam)}$= 15.589X 105 kJ/day

b. Energy Balance on Dryer: the wet chalk was dried at temperature of 120^oC.

H: Enthalpy (kJ/hr);

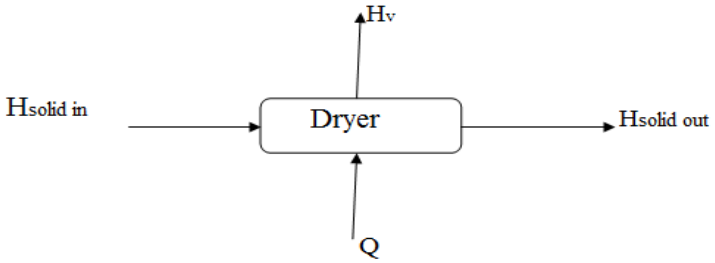


Figure 24: Energy Balance on Dryer

Q: the total amount of heat to be supplied (kJ/hr);
Mw: Mass flow rate (kg/hr);
Cp: specific heat (kJ/(K.kg)); T: Temperature (K); To = 100K: initial temperature;
Tr = 298K: reference temperature.
H(solid in) + Q = H_v + H(solid out)
11,478 + 53,887 + Q = 388,032 + 14,497
Q = 3.372 x105 kJ/hr

5.4 Design of Equipment

The main objective of this design is to determine the total heat transfer surface area, diameter, length/height and wall thickness of the equipment and material for construction.

a) Dimension of the Kettle Calciner

Mass feed rate of CaSO_4.H_2O = 3025.38 kg/hr and density = 2320 kg/m^3
Volumetric flow rate of CaSO_4.H_2O = = 1.305 m^3/hr

The time it takes for one batch is 2 hours and 30 minutes
The volume of the kettle = Volumetric flow rate of the CaSO4.H2O * time = 1.305 m^3/hr *2.5 hrs = 3.7625m^3
Assuming 15% safety margin m^3 = 3.7625 * 0.15 = 0.564m^3
Therefore total volume of the kettle = 3.7625m^3 + 0.564 m^3 = 4.33m^3
From the geometry of material with cylindrical form, the volume = $\pi(R^2)$h From physical space of the plant let the height of the kettle be 2.2m. Then, the radius of the kettle can be calculated as:
4.33m^3 = $\pi(R^2)$h => 4.33m^3 = 3.14* 2.2m R^2 => R^2 = 0.63 => R = 0.81m
Diameter of the mixer, D = 2R = 1.62m

b) Dimension of the Mixer

Mass feed rate of CaSO_4.H_2O = 2453.54 kg/day = 154 kg/hr and density = 2320kg/m^3
Mass feed rate of H_2O = 2944.25kg/day = 184 kg/hr and density = 1000kg/m^3
Volumetric flow rate of CaSO_4.H_2O = 0.066m^3/hr
Volumetric flow rate of H_2O = 0.184m^3/hr
Volumetric flow rate of the mixture = 0.066 m^3/hr + 0.184m^3/hr = 0.25 m^3/hr
The time it takes in one mixture is 4 minutes
The volume of mixer = Volumetric flow rate of the mixture * mixture time = 0.25m^3/hr * 4minutes = 0.017m^3
Assuming 15% safety margin m^3 = 0.017 * 0.15 = 0.00255m^3
Therefore total volume of the mixer = 0.017 m3 + 0.00255m^3 = 0.02m^3
From the geometry of material with cylindrical form, the volume = $\pi(R^2)$h
From physical space of the plant let the height of the mixer be 0.8 m.
Then, the radius of the mixer can be calculated as

$1.2m^3 = \pi(R^2)$h => $0.02m^3 = 3.14R^2$ => R^2 = 0.008 => R = 0.089m
Diameter of the mixture, D = 2R = 0.18m

c) Dimension of the Moulder

Mass feed rate of slurry = 5387 kg/day = 337kg/hr and density = 2757kg/m^3
Volumetric flow rate of slurry = $0.122m^3$/hr
The time it takes in one moulding is 15 minutes The volume of the moulder = Volumetric flow rate of the moulder * mixture time= $0.122m^3$/hr * 15 minutes = $0.0306m^3$
Assuming total 24% safety margin m^3 = 0.0306 * 0.24 = $0.00744m^3$ Therefore total volume of the moulder = $0.0306m^3$ + $0.0074m^3$ = $0.038m^3$
Volume = $\pi(R^2)$h and let the height of the moulder be 1m.
Then, the radius of the moulder can be calculated as
0.038 $m^3 = \pi(R^2)$h => 0.038 m^2 = $3.14R^2$ => R^2 = 0.013 => R = 0.113m
Diameter of the moulder, D = 2R = 0.226m
From the literature review, diameter and length of piece of chalk is 10mm and 80mm respectively.
Volume of the chalk = $0.00005m^3$
Number of chalk per hour = 756 pieces/hr = 12096 pieces/day

5.5 Financial Analysis

Experimentally and engineering wise it has been seen that investing chalk production for the plant is feasible. But, its feasibility should be checked financially. In this section, the proposed project to be analyzed financially depending on the proposed and designed chalk production plant according to [74].

5.5.1 Total Capital Investment

The total capital investment (TCI) for the production of blackboard chalk is estimated by summing up the fixed capital investment (FCI) and working capital. The working capital (WC) is taken as 15% of the total capital investment (0.15TCI) which is commonly used [75].
TCI = FCI + WC

a. Machinery and equipment The machinery and equipment cost of the chalk was calculated in table 31 below and the total estimated cost is 6,794,182.75 Birr.

b. Direct and Indirect cost: both the direct and indirect cost of the plant determination is done based on the ratio of solid-fluid processing plant because of in the production of blackboard chalk these processes are included [74].

WC = 15% of TCI = 0.15TCI By substituting this equation in equation above, we get the following value:
TCI = FCI +WC = FCI + 0.15 TCI => 0.85 TCI = FCI
=>0.85TCI = 17,423,681.78

Table 28: Purchased equipment cost

S.N	Description	Quantity	Unit Price(Birr)	Total Cost(Birr)
1	Jaw crusher	1	1,012,888	1,012,888.00
2	Secondary crusher	1	886,198	886,198.00
3	Packing/ bagging			930,576.00
4	Electric control panel			747,114.00
5	Spare parts			410,356.00
6	Weighing scale	2	15,000	30,000.00
7	Moulding	20	45,952	919,042.00
8	Dryer	1	971,811	971,811.00
	Contingencies		15%	886,197.75
	Total PEC			**6,794,182.75**

Table 29: Fixed Capital Investment Cost

Cost Category	Components	Factors	Cost (Birr)
	Purchased Equipment Cost	%PEC	6,794,182.75
Direct Cost (DC)	Equipment installation	35	2,377,964.00
	Instrumentation and controls	10	679,418.28
	Piping (installed)	30	2,038,254.83
	Electrical (installed)	10	679,418.28
	Buildings (Including services)	20	1,358,836.55
	Yard improvements	10	679,418.28
	Services facilities (installed)	40	2,717,673.10
	Land	4	271,767.31
	Total direct cost (DC)		**10,802,750.63**
Indirect Cost (IC)	Engineering and supervision	30	2,038,254.83
	Construction expenses	34	2,310,022.14
	Total Indirect cost (IC)		**4,348,277.00**
	DC + IC		**15,151,027.63**
Contractor's fee (CF)		5% (DC + IC)	757,551.38
Contingency (C)		10% (DC + IC)	1,515,102.76
FCI	DC + IC CF +C		**17,423,681.78**

=>TCI = 20,498,449.14
WC = 0.15 * 17,423,681.78 = 3,074,767.37

5.5.2 Total Product Cost

The total production cost for the production of board chalk is estimated by summing up the manufacturing cost and general expenses. The manufacturing costs can be calculated by adding direct production cost, fixed charge and plant overhead cost and the general expenses also calculated by summing up the distribution and marketing expenses, research and development, financing and gross earnings expenses [76].
Total Product Cost = Manufacturing Cost + General Expenses
Manufacturing Cost = direct production cost + fixed charges + plant overhead costs
General expenses = administrative costs + distribution and selling costs + research and development costs

1. Manufacturing Cost The different type of product cost under manufacturing are calculated as follows:

I. Direct Production Costs (DPC)

Table 30: Raw and auxiliary materials, Utilities cost and Operating Labor Cost

S.N	**Description**	**Unit**	**Unit Price (Birr)**	**Total Cost (Birr)**
	A. Raw and	**Auxiliary**	**Materials**	
1	Gypsum rocks	m^3	43.00	24,295.00
2	Process water	m^3	3.00	318.00
3	Diesel oil	lt	4.50	28,125.00
4	Plastic bag	pc	1.30	2,512.90
5	Kerosene	lt	3.67	128,101.35
6	Lubricating oil	lt	13.75	17,751.25
7	Cola	Kg	10.77	3,304.24
8	Carton box	pc	0.33	44,384.34
	Total			**247,792.08**
	B. Utility Cost			
1	Electric power	kWh	0.34	36,007.02
2	Water	m^3	3.00	4,758.00
	Total			**40,765.02**
	C. Operating	**Labor**	**Cost(OLC)**	
S.N	Job Title	Persons	Monthly(Birr)	Annually(Birr)
1	Project manager	1	4,000	48,000
2	Secretary	1	1,300	15,600
3	Documentation clerk	1	600	7,200
4	Accountant and controller	1	1,500	18,000
5	Accounts clerk	1	600	7,200
6	Cashier	1	800	9,600
7	Purchaser	1	800	9,600
8	Store keeper	1	800	9,600
9	Driver	1	600	7,200
10	Production	20	21,700	260,400
11	Guard	2	1,750	21,000
12	Cleaner	1	450	5,400
	Total	33	34,900	418,800
	Employee's Benefit	(25% of Basic	Salary)	104,700
	Total			**523,500.00**

D. Maintenance and repairs = 4%FCI = 0.04*17,423,681.78 = 696,947.27

E. Laboratory charges = 12% of operating labor = 0.12*523,500 = 62,820.00

F. Operating Supplies =11% of maintenance and repairs = 0.11*696,947.27 = 76,664.20

G. Direct supervisory and clerical labor =15% of operating labor = 0.15*523,500 = 78,525.00

Total direct production cost (DPC) = A + B + C + D + E + F + G

DPC = 247,792.08 + 40,765.02 + 523,500.00 + 696,947.27 + 62,820.00 + 76,664.20 + 78,525.00

DPC = 1,727,013.57 Birr/yr

II. Fixed Charges (FC)

H. Depreciation = 10% FCI = 0.1*17,423,681.78 = 1,742,368.18

I. Capital charge = 1.5% FCI = 0.015*17,423,681.78 = 261,355.23

J. Insurance = 0.5% FCI = 0.005*17,423,681.78 = 87,118.41

Fixed charges = FC = H + I + J = 1,742,368.18 + 261,355.23 + 87,118.41 FC = 2,090,841.82 Birr/yr

III. Plant Overhead Costs (POC)

Plant overhead costs = POC = 10% TPC = 0.1TPC

Manufacturing Cost (MC) = DPC + FC + POC = I + II + III

= 1,727,013.57 + 2,090,841.82 + 0.1 TPC = 3,817,855.39 + 0.1 TPC

1. General Expenses K. Administrative cost = 4% TPC = 0.04TPC

L. Distribution and selling cost = 10% TPC = 0.1TPC

M. Research and Development cost = 5% TPC = 0.05TPC

General Expenses= K + L + M = 0.04 TPC + 0.1TPC + 0.05TPC = 0.19 TPC

Total Production Cost (TPC) = Manufacturing cost + General expenses

TPC = 3,817,855.39 + 0.1 TPC + 0.19 TPC => 0.29 TPC = 3,817,855.39

TPC = 13,165,018.58 Birr/yr

5.5.3 Profitability Measurements

Annual production of chalk = Q = 2240 kg/day x 300 days/yr = 672,000 kg/yr

$$\mathbf{Unitproductcost} = \mathbf{UPC} = \frac{\mathbf{Totalproductioncost}}{\mathbf{Annualproduction}} = 16.59 Birr/kg \tag{4}$$

Selling price with 25% profit = SP = 0.35*16.59 + 16.59 = 22.4 Birr/kg

Total income per year (TI) = annual production rate * selling price = Q*SP = 672,000 * 22.4 = 15,050,448.00 Birr/yr

a. Gross profit earning (GPE): is the difference between sales and the cost of goods sold [74].

GPE = Total Income (TI) - Total Product Cost (TPC)

= 15,050,448.00 - 13,165,018.58 = 1,885,429.42 Birr/yr

Net profit earning (NPE): taking the income tax rate of Ethiopia which is 35%,

NPE = GPE - Income tax (0.35*GPE) = GPE (1-0.35) = 0.65GPE

= 0.65 * 1,885,429.42 = 1,225,529.12 Birr/yr

b. Return on investment (ROI): measures the gain or loss generated on an investment relative to the amount of money invested [74].

$$\mathbf{ROI} = \frac{\mathbf{NPE} * \mathbf{100}}{\mathbf{TCI}} = 6\% \tag{5}$$

c. Payback Period (PBP): It is a period required to recover the original investment outlay through the accumulated net cash flows earned by the project[75].

$$\mathbf{PBP} = \frac{\mathbf{FCI}}{\mathbf{NPE} + \mathbf{Depreciation}} = 3.6 years \tag{6}$$

d. Break Even Analysis: The break-even analysis is to determine the point at which sales revenues equal with the costs of products sold. When sales are below this point, the plant is making a loss, and at the point where revenues equal to costs, the plant is breaking even. The break-even production is the number of units necessary to produce and sell in order fully to cover the annual fixed costs [76]. It can be computed as:
Total product cost = Total income
To calculate the total product cost and total income, unit product cost of 13.7 birr/kg, product-selling price of 18.5 birr/kg, were considered. The break-even point was calculated as [77]:
(Fixed charges + General expenses + Plant overhead costs) + 16.59n = 22.4n
Where n is the number of kg of product produced.
2,090,841.82 + 0.1TPC + 0.19 TPC + 16.59n = 22.4n
2,090,841.82 + 3,817,855.39 = 5.81 n
5,908,697.21 = 5.81n => n = 1,016,987.47 kg

This is the quantity of product at break-even point. It is 52% of the plant capacity. Based on the preliminary economic evaluation, the suggested project has a return on investment (ROI) of 6% and payback period of 3.6 years. The break-even production capacity is at 52%, showing that there is good profit margin. Thus, the suggested project is financially feasible.

5.6 Plant Location

To select proper locations and sites, certain key requirements or criteria should be set (i.e. raw material proximity, Infrastructures, product market, human power) that would allow the assessment of a number of potential locations. The remaining alternatives are subject to a more in-depth qualitative and quantitative analysis of technical and financial criteria, including social, environmental and economic aspects of location and site selection. For gypsum extract producing plant, based on proximity to market, availability of utilities such as water, electricity and other infrastructures, ease of access and handling of human resource, around Abay was selected to locate the plant.

6 CONCLUSION AND RECOMMENDATION

6.1 Conclusion

Gypsum was taken from different sources and characterized both physically and chemically. Physical characterization of the sample shows that:

- it is so soft (its hardness is 2.0 Moh's scale) that a fingernail can scratch it and
- it is colorless, white, gray, brown, orange, pink, yellow, light red and green.

Chemical characterization of the sample shows that its compositional analysis:

- 37.4% of CaO, 49.98% of SO_3 and 11.03% of H_2O for Kentry gypsum;
- 38.23% of CaO, 50.43% of SO_3 and 11.03% of H_2O for Blue Nile Gorge gypsum;
- 30.75% of CaO, 44.57% of SO_3 and 16.87% of H_2O for Yogef (Lower gypsum bed) gypsum and
- 30.99% of CaO, 45.61% of S$O_3$and 18.67% of H_2O for Yogef (Upper gypsum bed) gypsum.

All the compositional analysis show that the gypsum quarried from country were best for chalk production.
In this study, the bulk density and breaking strength in production of quality chalk was modeled and analyzed through CCD of RSM Design Expert 7.0 version. Particle sizes, calcinations temperature and moisture content have been employed to carry out the experimental study. The data was suitably used to explore two factor interactions and quadratic response surfaces for bulk density and breaking strength respectively and construct second order polynomial models.

The results show that experimental data and data obtained by regression equation closely correlate each other, which validate the regression equations developed. Therefore, the present study has shown that RSM combined with a CCD provides a very reliable and accurate methodology for optimizing experiments for the production of quality chalk from gypsum. Analysis of variance was also found to be significant for both the responses. The predicted values match with the experimental values reasonably well, with R^2 of 0.9991 for bulk density and R^2 of 0.9996 for breaking strength.

The Design-Expert software used searches for a combination of factor levels that simultaneously satisfy the requirements placed on each of the responses and factors. Optimization requires that goals (i.e., none, maximum, minimum, target, or in range) are set for the variables and response where all goals were combined into one desirability function. To find a good set of conditions that meets all the goals, the three variables: particle

size (45μm - 95μm) and moisture content (5% - 7%) were set within range and calcinations temperature (135^oC - 145^oC) was minimized within range while the responses bulk density and breaking strength were set in a target.

The mathematical model developed could predict the bulk density and breaking strength at any point inNPE*100 the experimental domain as well as the determination of the optimal process parameters. The high correlation in the model indicates that the second order polynomial model could be used to optimize the responses. Formulation with highest desirability (D = 0.998) was selected as optimum. The optimal value obtained 1.1445g/cm^3 and 13.85N/cm respectively for bulk density and breaking strength were found to be 85.55μm particle size, 137^oC for calcinations temperature and 6.25% for moisture content as optimum value of process parameters. These results implicate that the optimization using a RSM based on CCD was useful software in improving the optimization of quality chalk parameters.

6.2 Recommendation

It is recommended that to use gypsum raw from Derba Mugher site (kentry) as a first priority.

- Because of the quality of gypsum occurrences fulfils the required standard for chalk and gypsum manufacturing, which was confirmed by chemical analysis.
- The supply of raw material from Kentry is adequate enough in the future, as the resources of Kentry gypsum is in the range of millions of tons.
- Relatively lower transportation cost is incurred as compared to the other gypsum occurrences.
- As the supply of the raw materials is in a crushed by the Mugher Cement Enterprises, the cost energy to crush at the factory will be proportionally reduced.

There are many factors that affect the quality of chalk but this thesis study considered only three factors (particle size, calcinations temperature and moisture content), it is possible to test the other factors such as calcinations time, kerosene to oil ratio of lubricate the moulding and drying time.

This paper also recommends as calcinations kettle can be changed from batch to continuous and it was used oven dryer for dry a chalk after moulding, it is possible also to dry by solar without any cost. The obtained quality of chalk was the optimum one, so if any company was design a factory and work accordingly, by the same selling price with the existing chalk, it is still feasible.

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APPENDICES

Table 31: Experimental Design Run Sheet

Parameters	**Run 1**	**Run 2**	**Run 3**	**Run 4**	**Run 5**
Particle Size (μm)	45	70	95	70	70
Calcinations Temp (o^C)	140	140	135	135	145
Moisture Content (%)	6	6	7	6	7
Bulk Density (g/cm^3)	1.206	1.150	1.104	1.166	1.125
Breaking Strength (N/cm)	13.2	14.0	12.0	13.8	11.8
Parameters	**Run 6**	**Run 7**	**Run 8**	**Run 9**	**Run 10**
Particle Size (μm)	70	95	70	45	70
Calcinations Temp (o^C)	145	145	140	145	145
Moisture Content (%)	6	7	7	5	5
Bulk Density (g/cm^3)	1.135	1.064	1.145	1.193	1.140
Breaking Strength (N/cm)	14.3	12.6	11.5	15.9	16.6
Parameters	**Run 11**	**Run 12**	**Run 13**	**Run 14**	**Run 15**
Particle Size (μm)	95	95	95	70	45
Calcinations Temp (o^C)	135	145	140	140	140145
Moisture Content (%)	5	5	6	5	7
Bulk Density (g/cm^3)	1.120	1.084	1.093	1.154	1.18
Breaking Strength (N/cm)	17.0	17.5	14.9	16.4	10.8
Parameters	**Run 16**	**Run 17**	**Run 18**	**Run 19**	**Run 20**
Particle Size (μm)	45	95	95	45	45
Calcinations Temp (o^C)	140	140	145	135	135
Moisture Content (%)	5	7	6	7	5
Bulk Density (g/cm^3)	1.210	1.088	1.075	1.214	1.225
Breaking Strength (N/cm)	15.7	12.4	15.2	10.2	15.5

Table 32: Design Summary of Designs Expert

Study Type	Response Surface
Initial Design	Central Composite
Design Model	Quadratic Polynomial
Runs	20
Block	No Block

Table 33: Regression Coefficient of 2FI and Significance for Bulk Density

Factors	**Coefficient Estimate**	**95% CI Low**	**95% CI High**
Intercept	1.15	1.15	1.15
A - PS	-0.056	-0.057	-0.054
B - CT	-0.017	-0.018	-0.016
C - MC	-7.00×10^{-3}	-8.014×10^{-3}	-5.985×10^{-3}
AB	-1.385×10^{-3}	-2.640×10^{-3}	-1.138×10^{-4}
AC	-1.695×10^{-3}	-2.895×10^{-3}	-4.955×10^{-4}

Table 34: Regression Coefficient of quadratic and Significance for Breaking Strength

Factors	**Coefficient Estimate**	**95% CI Low**	**95% CI High**
Intercept	14.06	14.02	14.12
A - PS	0.84	0.80	0.87
B - CT	0.26	0.23	0.30
C - MC	-2.51	-2.54	-2.47
AC	0.071	0.033	0.11
BC	0.051	0.013	0.089
C^2	-0.12	-0.18	-0.063

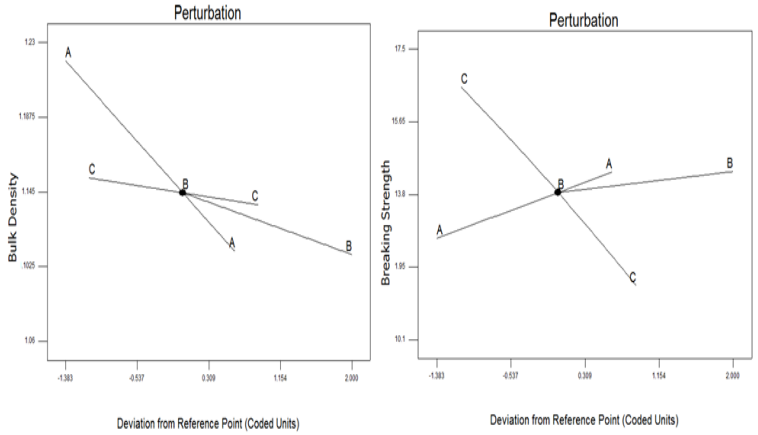


Figure 25: Perturbation plots for (i) bulk density and (ii) breaking strength

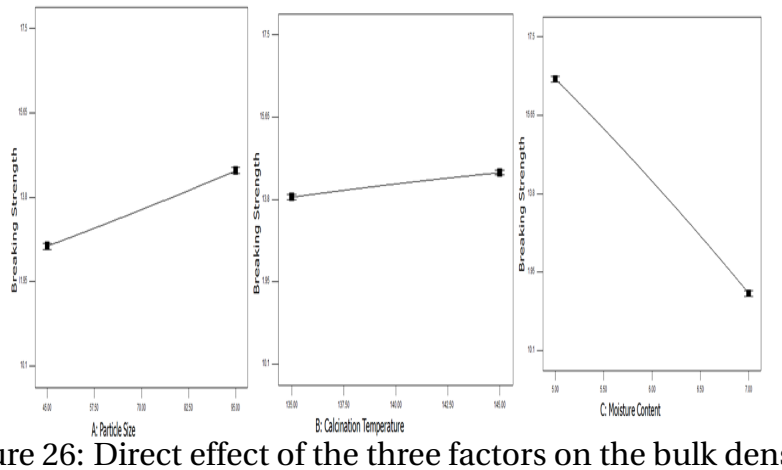


Figure 26: Direct effect of the three factors on the bulk density

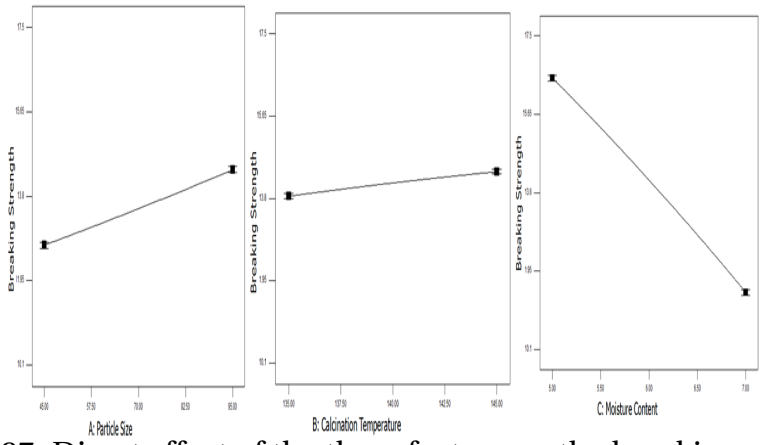


Figure 27: Direct effect of the three factors on the breaking strength

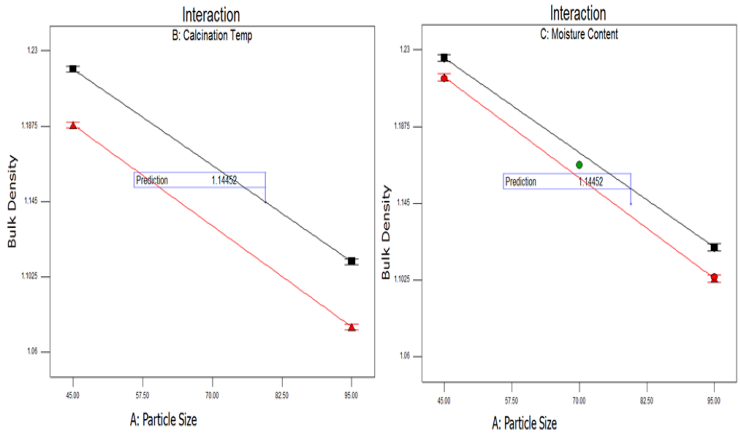


Figure 28: Effect of two factor interactions on bulk density

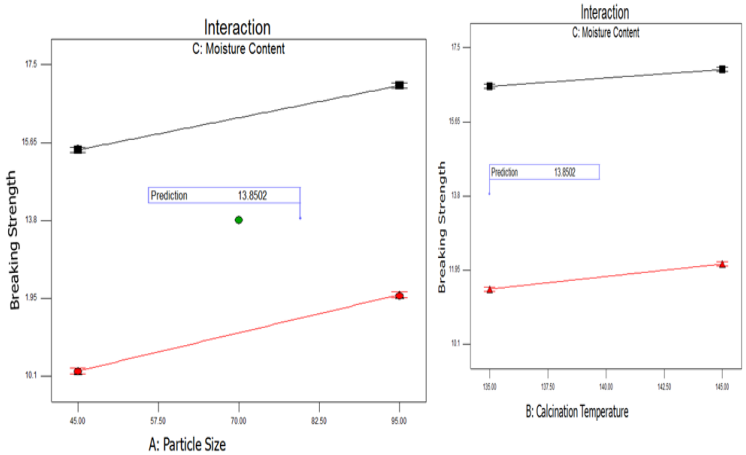


Figure 29: Effect of two factor interactions on breaking strength

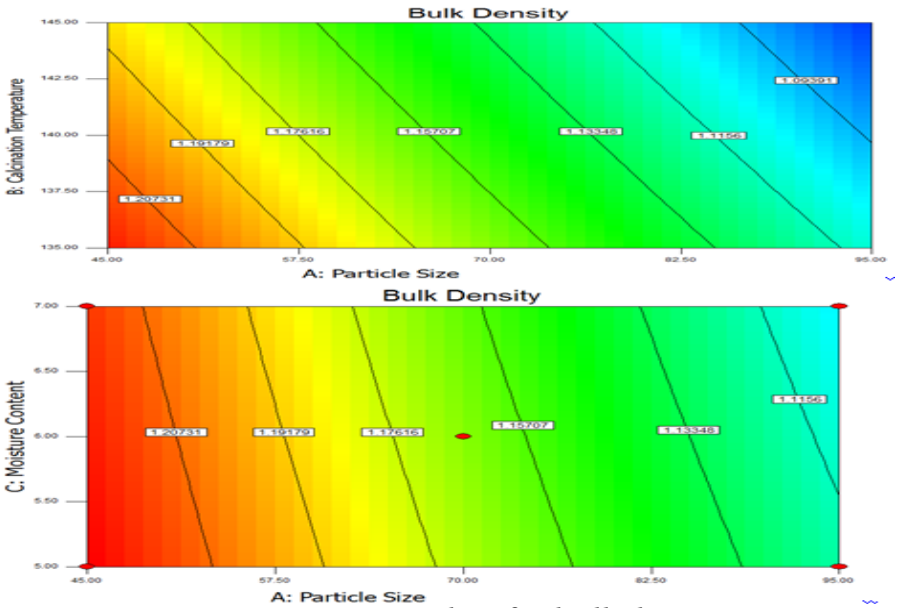


Figure 30: Contour plots for bulk density

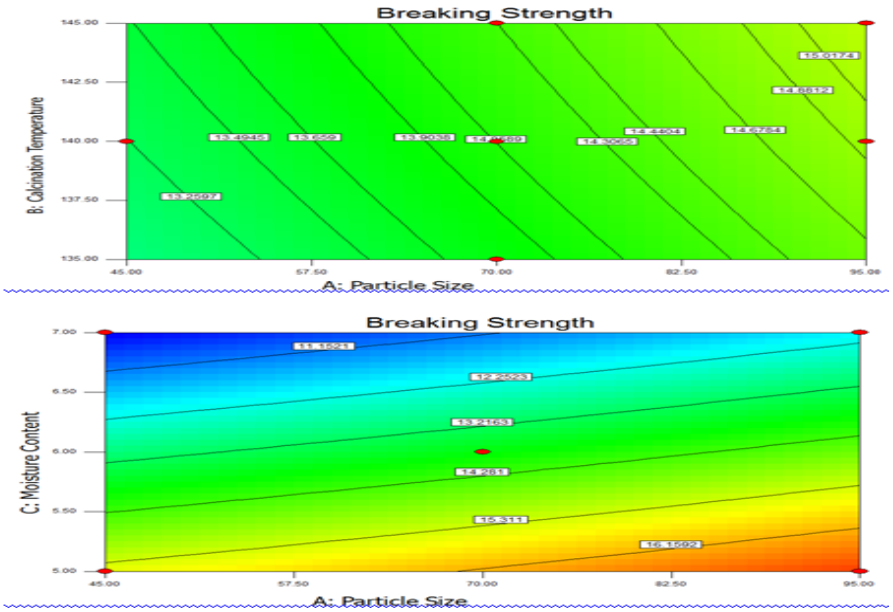


Figure 31: Contour plots for breaking strength

Declaration

I, **the undersigned**, declare that this thesis entitled "**Optimization of Gypsum Processing Parameters For the Production of Improved Quality Chalk**" has not been submitted in any form for another degree, diploma or an award at any university or other institution of the tertiary education. Whenever contributions of others are involved, every effort is made to indicate this clearly, witH due references to the literature review and discussions. Iformation taken from the published and unpublished work of the others has been acknowledged in the text and a list of references was give.

The work was under the guidance of Dr. Tassisa Kaba (Asst. Prof.) instructor in Addis Ababa University, Addis Ababa Institute of Technology, School of Chemical and Bio Engineering.

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Name

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Signature

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Place of Submission

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Date of Submission

Certification

This thesis has been read and approved as meeting the requirement of the School of Chemical and Bio Engineering in the Addis Ababa Institute of Technology for the award of degree of masters of Science in chemical engineering.

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Advisor

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Signature

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Date