A COMPARATIVE ANALYSIS OF
THE RECENT CEMENT GRINDING SYSTEMS
WITH PARTICLE-BASED INFLUENCES ON CEMENT PROPERTIES

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ABSTRACT

A COMPARATIVE ANALYSIS OF THE RECENT CEMENT GRINDING SYSTEMS WITH PARTICLE-BASED INFLUENCES ON CEMENT PROPERTIES

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The conventional cement grinding system, the ball mill, has very poor efficiencies in spite of innovative improvements. For this purpose, development of new techniques, which allow proper size reduction and uniform particle size distribution with less specific energy consumptions, have become a necessity.

The aim of this study is to make a comparative analysis of the fairly new cement grinding technologies, COMFLEX® Grinding System, Roller Press and HOROMILL®, at the same cement production plant with the same raw materials.

In this context, CEM I 42.5 R type cement was produced with a fixed Blaine fineness of 3600 (±100) cm²/g at three different grinding units. The same raw materials, clinker and gypsum, and identical feeding ratios, 95% and 5%, were used to produce cement. Accordingly, these different grinding techniques were inspected with respect to the microstructural properties of cement particles, and the relative chemical, physical and mechanical properties of products.

It was found that the main cement grinding parameters, specific surface area and sieve residue, do not show expected relation and change with each grinding system due to differences in the size reduction technique. Moreover, strength and other hardened mortar properties are directly affected by the liberation conditions of reactive grains at grinding stages.
High capacity and low specific energy consumption i.e. the breaking and cracking efficiency of the roller press and higher grinding performance of the ball mill promoted the COMFLEX® system. On the other hand, the roller press was clearly advantageous at early strength performances with moderate specific energy usages during grinding. Nonetheless, it also had drawbacks like higher water demand and earlier setting times (which mean higher hydration temperatures). When the wideness and sharpness of classification results were considered, HOROMILL® gave better results with high circulation and efficient air classification design; although there were weaknesses of the system such as lower capacity and higher specific energy consumption rate.

Keywords: Cement Grinding, COMFLEX, HOROMILL, Roller Press, Size and Shape of Cement Particles
ÖZ

YENİ ÇİMENTO ÖĞÜTME SİSTEMLERİNİN KARŞILAŞTIRMALI ANALİZİ İLE TANECİK YAPISI BAZINDA ÇİMENTO ÖZELLİKLERİNE ETKİLERİ

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Geleneksel çimento öğütme sistemi olan bilyalı değirmenler, çeşitli geliştirme çalışmalarına rağmen öğütme verimliliği düşük sistemlerdir. Bu sebeple uygun tane boyu küçültülebilirliği ve düzenli tane boyu dağılımını daha düşük birim enerji sarfiyatları ile sağlayabilecek yeni sistemlerin geliştirilmesi zorunluluk haline gelmiştir.

Bu tez çalışmasında yeni tipteki öğütme sistemlerinden COMFLEX® öğütme sistemi, Roller Press ve HOROMILL®’in aynı çimento üretim tesisi ve bu tesisin hammaddelerini karşılaştırmalı değerlendirmelerinin yapılması amaçlanmıştır.

Bu kapsamda, CEM I 42,5 R tipi çimentonun sabit 3600 (±100) cm²/gram yüzey alanı değeri ile üç farklı öğütme sistemi kullanılarak üretimi gerçekleştirilmiştir. Aynı özellikteki klinker ve alçı hammaddeleri %95 ve %5 oranlarında kullanılarak üretim yapılmıştır. Dolayısıyla bu farklı öğütme teknikleri, çimento tanelerinin mikro yapısal özellikleri ve beraberinde kimyasal, fiziksel ve mekanik özellikleri açısından incelenmiştir.

Ana çimento öğütme parametrelerinden birim yüzey alanı ve elek bakiyesi belirgin bir ilişki göstermemekte, farklı öğütme prensiplerine bağlı olarak her sistemde farklı sonuçlar ortaya çıkmaktadır.

Anahtar Kelimeler: Çimento Öğütme, COMFLEX, Horomill, Roller Press, Çimento Tane Boyu ve Yapısı
To My Wife...
I would like to express my gratitude to my supervisor Prof. Dr. Çetin Hoşten for his supervision, support, advice and patience throughout this study; and for his guidance and encouragement to study mineral processing since my undergraduate years in the Mining Engineering program and, then, in the Cement Engineering program.

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# TABLE OF CONTENTS

ABSTRACT ............................................................................................................. iv  
ÖZ ............................................................................................................................ vi  
ACKNOWLEDGMENTS .......................................................................................... ix  
TABLE OF CONTENTS ......................................................................................... x  
LIST OF TABLES ................................................................................................... xiii  
LIST OF FIGURES ................................................................................................ xiv  

<table>
<thead>
<tr>
<th>CHAPTERS</th>
</tr>
</thead>
<tbody>
<tr>
<td>1. INTRODUCTION</td>
</tr>
<tr>
<td>1.1 General</td>
</tr>
<tr>
<td>1.2 Objective and Scope of the Thesis</td>
</tr>
<tr>
<td>2. THEORETICAL CONSIDERATIONS</td>
</tr>
<tr>
<td>2.1 Portland Cement</td>
</tr>
<tr>
<td>2.2 Hydration of Cement</td>
</tr>
<tr>
<td>2.3 Cement Grinding Process</td>
</tr>
<tr>
<td>2.3.1 Cement Grinding Technologies</td>
</tr>
<tr>
<td>2.3.1.1 Ball Mill</td>
</tr>
<tr>
<td>2.3.1.2 Roller Press</td>
</tr>
<tr>
<td>2.3.1.3 Combined (Hybrid) Grinding System</td>
</tr>
<tr>
<td>2.3.1.4 Horizontal Roller Mill (HOROMILL®)</td>
</tr>
<tr>
<td>2.3.2 Classification Systems</td>
</tr>
<tr>
<td>2.3.2.1 First Generation Separators</td>
</tr>
<tr>
<td>2.3.2.2 Second Generation Separators</td>
</tr>
<tr>
<td>2.3.2.3 Third Generation Separators</td>
</tr>
<tr>
<td>2.3 Effects of Grinding Techniques on Specific Energy Consumptions</td>
</tr>
<tr>
<td>2.4 Effects of Particle Size Distributions and Microstructural Properties on the Performance of Cement</td>
</tr>
<tr>
<td>3. EXPERIMENTAL STUDY</td>
</tr>
<tr>
<td>3.1 Materials Used in the Study</td>
</tr>
<tr>
<td>3.2 Raw Materials</td>
</tr>
<tr>
<td>3.2.1 Clinker</td>
</tr>
<tr>
<td>3.2.2 Gypsum</td>
</tr>
<tr>
<td>3.2.3 Other Materials</td>
</tr>
<tr>
<td>3.3 Testing Equipment and Standards</td>
</tr>
</tbody>
</table>
3.4 Cement Grinding Systems ..................................................25
  3.4.1 COMFLEX® Grinding System ........................................25
  3.4.2 Roller Press System ..................................................26
  3.4.3 HOROMILL® Grinding System .......................................27

3.5 Experimental Program ...................................................28

3.6 Industrial Operations and Sampling Periods .........................29
  3.6.1 COMFLEX® Grinding System (Roller Press + Ball Mill) ......29
  3.6.2 Roller Press System ..................................................30
    3.6.2.1 Samples and Sampling Points ..................................30
  3.6.3 HOROMILL® System ..................................................30
    3.6.3.1 Samples and Taken Points .....................................31

3.7 Particle Size Distribution Analysis and Expression Methods ....31

3.8 Particle Shape Analysis ..................................................32

3.9 Physical and Chemical Quality Analyses of Products .............33
  3.9.1 Sieving and Surface Area Analysis ...............................33
  3.9.2 Compositional Analysis ............................................34
  3.9.3 Mortar Analyses ....................................................35
    3.9.3.1 Preparation of Mortars and Prisms ...........................36

3.10 Operational Efficiencies of Grinding Systems ....................37
  3.10.1 Classification Efficiency ..........................................37
  3.10.2 Energy Efficiency ..................................................39

4. RESULTS AND DISCUSSION ................................................40
  4.1 Particle Shape Characteristics .......................................40
  4.2 Particle Size Distributions ............................................43
  4.3 Quality Characteristics of Products ..................................44
    4.3.1 Chemical and Physical Characteristics ..........................44
    4.3.2 Compressive Strengths ............................................48
  4.4 Grinding Efficiencies of Systems ....................................49
    4.4.1 Operational Experiences ..........................................49
    4.4.2 Classifying Efficiency ............................................50
    4.4.3 Specific Energy Consumptions ...................................51
      4.4.3.1 COMFLEX® Grinding System ................................51
      4.4.3.2 HOROMILL® Grinding System .................................53
      4.4.3.3 Roller Press System ..........................................54

5. CONCLUSIONS .............................................................56

6. RECOMMENDATIONS ........................................................58

REFERENCES ........................................................................59
APPENDICES

A. PARTICLE SIZE ANALYSES OF PRODUCTS .................................................62
B. PARTICLE SIZE ANALYSES OF TAILINGS .................................................67
C. ROSIN RAMMLER DISTRIBUTIONS OF PRODUCTS ........................................71
D. TROMP CURVES FOR GRINDING SYSTEMS ..............................................73
E. SEPARATOR PERFORMANCE AND EFFICIENCY CURVES ...............................74
F. SCANNING ELECTRON MICROSCOPY ANALYSES OF PRODUCTS .................78
G. SAMPLING POINTS AT GRINDING SYSTEMS .............................................82
H. SEM ANALYSES OF CLINKER .....................................................................88
I. FLOWSHEETS OF GRINDING SYSTEMS ....................................................90
J. DESIGN PARAMETERS OF GRINDING SYSTEMS .........................................93
LIST OF TABLES

TABLES

Table 2.1 The main clinker phases................................................................. 5
Table 2.2 Average chemical composition of OPC clinker ....................... 5
Table 2.3 Amount of belite spots embedded in alite crystals .................21
Table 3.1 Chemical and Physical Analyses of Ordinary Portland Cement Clinker Samples ......................................................... 23
Table 3.2 Main Clinker Phases and Other Components of the Test Samples................................................................. 23
Table 3.3 Composition of Gypsum .................................................................23
Table 3.4 Cement Tests, Equipment and Related Standards ................. 24
Table 3.5 Size Distribution of CEN Standard Testing Sand ................. 36
Table 4.1 Mineralogical compositions of main phases in size fractions of grinding modes ................................................................. 42
Table 4.2 Amount of C₂S spots in C₃S crystals................................. 43
Table 4.3 XRF Analysis Results of Sampled Cements from Grinding Systems ......................................................................................... 44
Table 4.4 Clinker Quality Parameters, Phases and Other Chemical Analyses ......................................................................................... 45
Table 4.5 Physical and Mortar Performances of Sampled Products .... 45
Table 4.6 Compressive Strengths of Sampled Cements ...................... 48
Table 4.7 Equipment-Basis and Total Energy Consumptions for COMFLEX® Grinding System with Operational Parameters ............. 52
Table 4.8 Equipment Basis and Total Energy Consumptions for HOROMILL® System with Operational Parameters ...................... 53
Table 4.9 Equipment Basis and Total Energy Consumptions for Roller Press System with Operational Parameters ...................... 54
LIST OF FIGURES

FIGURES

Figure 2.1: Mechanical Parts of HOROMILL® ........................................11
Figure 2.2: Operating Principle of HOROMILL® ........................................12
Figure 2.3: Angles of nip at different grinding systems ..........................12
Figure 2.4: Laser particle size distributions of PC 42.5 R samples including varying addition amounts of −32+3 μm fraction .................17
Figure 2.5: Specific surface area values of PC 42.5 R samples with additions of varying −32+3 μm and −20+5 μm fractions ..................18
Figure 2.6: Compressive strength values of PC 42.5 R sample and different size fractions of that sample ........................................18
Figure 2.7: Compressive strengths of PC 42.5 R sample with additions of −32+3 μm, −20+5 μm and −30 μm size fractions .................19
Figure 2.8: Blaine and BET analyses .......................................................20
Figure 2.9: Liberation degree of Alite Crystals ......................................21
Figure 4.1: Sieve residues of 15 samples from different grinding systems...41
Figure 4.2: Blaine fineness of sieved 15 samples in the same order ............41
Figure 4.3: Dehydration behaviour of the gypsum that was used in the study ..............................................................................46
Figure 4.4: Developments of Compressive Strengths ................................49
CHAPTER 1

INTRODUCTION

1.1 General

It is the best way to understand the cement industry by considering the significant relation between cement consumption statistics and the level of economic development, as cement is mostly used in modern infrastructure and construction [1]. Thus, cement industry has grown with developing economies. In 1950, global cement production was less than 200 million tons; however, it has reached to more than 2500 million tons in 2006 [2]. Moreover, after 2010, global demand for cement is forecast to rise 4.1 % per year to 3500 million tons in 2013 [3]. Then, if 90 to 120 kWh/ton specific energy consumption in cement production is considered, it is obvious that the cement industry is an energy intensive industry. About 2% of global electrical energy is consumed by the cement industry, and 30 to 40% of the production cost of cement is accounted by electricity [4]. Although, electricity is consumed in all stages of the production process, 67% of total electrical energy is used by crushing and grinding operations. Again, 38% of total used individually by finished product (cement) grinding operations [5]. Therefore, cement grinding operations have been studied and improved consistently, and various designs have been practiced to enhance efficiency of the process.

Finish grinding is the single highest energy consuming operation in cement manufacturing. However, only 2 to 20% of the supplied energy goes to size reduction in the grinding system. A major part is lost as friction, sound, heat, vibration and also as other inefficiencies on mechanical transfers [6]. Therefore, grinding optimization becomes a continuous requirement to achieve higher efficiency from the system. Different grinding systems have been designed and operated to improve the process. In addition to the closed-circuit, two-compartment ball mills known for decades, combined grinding systems with closed-circuit high pressure grinding rolls and downstream closed-circuit ball mills have been used for nearly 20 years. The horizontal roller mill (HOROMILL®) is another relatively new grinding system having different compression and size.
reduction features [7]. Furthermore, nowadays, high pressure grinding rolls are singly used to produce cement, and the research to improve this mode of grinding has been continuing. The aim of this progress and using different modes of grinding is to achieve higher efficiencies and so as to decrease energy consumptions.

However, besides energy consumption, the quality of the produced cement is directly related with the mode of grinding. In addition to obtaining the required specific surface area, the grinding operation is to generate a particle size distribution suitable for obtaining the desired service performances from the cement. 3 to 30µm size fraction is important for strength development of cement. Below 3 µm, particles are only effective on initial strengths; and hydration is slow and less influential on strength above 60 µm [6]. Therefore, efficiency of classifiers in the grinding circuit is of importance also as they influence the product size distribution.

On the other hand, breakage mechanisms of various grinding systems - due to different forces acting on particles – cause different surface area characteristics. Larger surface area - because of microcracks on particles – enables better reactivity of crystals inside the clinker, and promotes higher strengths [8]. In addition, shapes of the particles are also effective on transporting and service properties of cement; like flowability of cement at dispatching, workability and water demand of concrete [9].

1.2 Objective and Scope of the Thesis

It is the objective of this thesis to compare three different grinding systems on the basis of grinding technologies with the effects on performances of products. CEM I 42.5 R type of cement is selected for the experimental study, because it is composed of only clinker (95%) and gypsum (5%), which are the main components for all types of cements. Furthermore, higher clinker ratio -without any other strength affecting component- gives clear performance results, and misconceptions due to diversity of materials are prevented.

The three grinding systems tested in this thesis study are: the COMFLEX® grinding system, the horizontal roller mill (HOROMILL®) and the roller press. Intermediate and final product streams of these grinding systems were sampled to evaluate size classification efficiencies and service properties of the final cement products. In addition, energy efficiency of the systems and micro-
structural properties of the ground cement products were also investigated by various means.

This thesis is composed of six chapters:

Chapter 1 gives an introduction about the importance of the grinding process in cement manufacturing and the requirements of innovations to improve efficiencies and consequences about products.

Chapter 2 presents the definition of Portland cement, manufacture of clinker and Portland cement, chemical composition of clinker and Portland cement, hydration of cement, grinding process in cement production with several designs, and classification units.

Chapter 3 presents the properties of materials used in the study, testing equipment and standards, properties of COMFLEX® Grinding, Roller Press and HOROMILL® systems, experimental procedures, sampling works in study at the production of CEM I 42.5 R, particle size distribution analysis and illustrations, particle shape analysis, physical and chemical quality analysis, mechanical strength tests, operational efficiencies of grinding systems.

Chapter 4 presents discussions about grinding systems and produced cements with respect to particle shape characteristics, particle size distributions, quality characteristics with chemical, physical and mechanical test results, and efficiencies of grinding systems.

Chapter 5 presents the conclusions of the study derived from the findings of the test productions and experimental results of produced cements.

Chapter 6 presents recommendations for future researchers.
CHAPTER 2

THEORETICAL CONSIDERATIONS

2.1 Portland Cement

Ordinary Portland Cement (OPC) is the most commonly used cement. This hydraulic binder was named after the island “Portland” in Great Britain and was patented by Joseph Aspdin in 1824. Joseph Aspdin gave that name because the color of the produced cement was the same as the color of the Portland stone (oolitic limestone) which was commonly used for buildings in this area. Aspdin crushed a hard limestone and calcined it, and mixed the lime with clay. He then applied wet grinding to that raw mix, and calcined the ground component in a kiln, CO$_2$ was expelled. At the end, main component of cement was obtained, and called “clinker” which was ground to get a powdered material, cement [10].

Modern Portland cement is manufactured in a series of processes. Calcium oxide (lime), silica, aluminum oxide and ferric oxide are the main components; and they have to be in suitable proportions to produce clinker. The source of lime is limestone (mainly calcium carbonate), and silica is obtained from clay which also supplies aluminum oxides and ferric oxides. However, bauxite (for Al$_2$O$_3$) and iron ores -like hematite, goethite etc.- (for Fe$_2$O$_3$) are also used if the presence of these oxides is not enough in the clay used. Properly proportioned material mix is crushed and ground to get the raw meal. Then the raw meal is heated in a kiln, firstly to decompose CO$_2$ and then to react the obtained calcium oxide with the other components to form calcium silicates and aluminates which form at various temperatures up to 1450 °C. At the end of the reactions in the kiln, rapidly cooled nodular clinker is obtained. There are four different clinker phases, as shown in Table 2.1. In addition to the main constituents, a number of other compounds (Na$_2$SO$_4$, K$_2$SO$_4$, MgO, and CaCO$_3$) are present in these clinker phases [11].
Table 2.1: The main clinker phases [11]

<table>
<thead>
<tr>
<th>Name of the pure mineral</th>
<th>Idealized chemical composition</th>
<th>Shorthand notation</th>
<th>Name of the mineral existent in the clinker</th>
<th>Average content in OPC [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>Tricalcium silicate</td>
<td>Ca$_3$SiO$_5$</td>
<td>C$_3$S</td>
<td>Alite</td>
<td>40 – 80</td>
</tr>
<tr>
<td>Dicalcium silicate</td>
<td>Ca$_2$SiO$_4$</td>
<td>C$_2$S</td>
<td>Belite</td>
<td>2 – 30</td>
</tr>
<tr>
<td>Tricalcium aluminate</td>
<td>Ca$_3$Al$_2$O$_6$</td>
<td>C$_3$A</td>
<td>Aluminate</td>
<td>3 – 15</td>
</tr>
<tr>
<td>Calcium aluminate ferrite</td>
<td>Ca$_2$AlFeO$_5$</td>
<td>C$_4$AF</td>
<td>Aluminate ferrite</td>
<td>4 – 15</td>
</tr>
</tbody>
</table>

(a) Key to cement shorthand notation: A = Al$_2$O$_3$, C = CaO, F = Fe$_2$O$_3$, H = H$_2$O, S = SiO$_2$
(b) Present as member of the solid solution series between Ca$_2$Fe$_2$O and Ca$_6$Al$_2$Fe$_5$O$_{16}$

The chemical composition of Ordinary Portland Cement clinker varies considerably because it depends on the chemical and mineralogical composition of the used raw materials and combustibles. The average chemical composition, as illustrated in Table 2.2, shows that the main components are CaO and SiO$_2$, and secondarily Al$_2$O$_3$ and Fe$_2$O$_3$ [12].

Table 2.2: Average chemical composition of OPC clinker [12]

<table>
<thead>
<tr>
<th></th>
<th>Average content in OPC [%]</th>
</tr>
</thead>
<tbody>
<tr>
<td>CaO</td>
<td>60 – 69%</td>
</tr>
<tr>
<td>SiO$_2$</td>
<td>20 – 25%</td>
</tr>
<tr>
<td>Al$_2$O$_3$</td>
<td>4 – 7%</td>
</tr>
<tr>
<td>Fe$_2$O$_3$</td>
<td>0.2 – 7%</td>
</tr>
<tr>
<td>MgO</td>
<td>0.5 – 5%</td>
</tr>
<tr>
<td>Na$_2$O + K$_2$O</td>
<td>0.5 – 1.5%</td>
</tr>
<tr>
<td>SO$_3$</td>
<td>0.1 – 1.3%</td>
</tr>
</tbody>
</table>

The setting of the cement paste is controlled by gypsum (calcium sulfate) addition at grinding stage of clinker. Calcium sulfate is a set regulator and prolongs the workability of the cement paste. Without additional sulfate, calcium aluminate hydrate (Ca$_4$[Al(OH)$_6$]$_2$(OH)$_2$·6H$_2$O) would be formed instantly after adding the mixing water to the cement which would cause a rapid setting of the cement paste and would abolish workability. In the presence of additional sulfate, ettringite (Ca$_6$[Al(OH)$_6$]$_2$(SO$_4$)$_3$·26H$_2$O) is formed on the surface of Ca$_3$Al$_2$O$_6$ during hydration which causes a delay of the otherwise rapid dissolution of Ca$_3$Al$_2$O$_6$ [12].
2.2 Hydration of Cement

In cement chemistry the reaction of unhydrated cement with water is called hydration which leads to chemical and physico-mechanical changes. Plasticity of the cement paste is quickly lost, and the cement paste hardens until the ultimate strength is achieved. Hydration of Portland cement and the hydration kinetics are affected by several conditions. The composition of phases in cement and amount of foreign ions like alkalis, the fineness characteristics of the cement such as particle size distribution and specific surface area, the water/cement-ratio of the mortar, curing conditions, and chemical additives (if used) [13].

Approximately 8 to 16 hours after the beginning of the hydration, alkali-sulfates dissolve quickly, and K, Na, and S are the effective components in the pore solution. Ca, hydroxide, and S concentrations are controlled by Ca(OH)\(_2\) and CaSO\(_4\) and the pore solution is highly oversaturated with respect to portlandite (Ca(OH)\(_2\)), gypsum (CaSO\(_4\cdot2\)H\(_2\)O), and ettringite (Ca\(_6\)[Al(OH)\(_6\)]\(_2\)(SO\(_4\))\(_3\)\(_2\)6H\(_2\)O) at early hydration times [14]. However, concentrations of Al, Fe, and Si are generally low in the pore solution. During the first hours the concentrations of these ions are generally limited because of the accumulation of initial hydrates around the clinker grains [15]. At later hydration times the ions in pore solution are limited by the following precipitating hydrates.

Siliceous clinker phases (C\(_2\)S, belite and C\(_3\)S, alite) are hydrated as;

\[
\begin{align*}
\text{Ca}_3\text{SiO}_5 + (3+m-n)\text{H}_2\text{O} & \leftrightarrow \text{Ca}_n\text{SiO}(2+n)\cdot m\text{H}_2\text{O} + (3-n)\text{Ca(OH)}_2 \\
\text{Ca}_2\text{SiO}_4 + (2+m-n)\text{H}_2\text{O} & \leftrightarrow \text{Ca}_n\text{SiO}(2+n)\cdot m\text{H}_2\text{O} + (2-n)\text{Ca(OH)}_2
\end{align*}
\]

The resultant calcium silicate hydrates are amorphous and have varying chemical compositions. According to that variance, they are described as C-S-H phases or C-S-H gel. CaO/SiO\(_2\) molar ratio of the formed C-S-H phases is 1.7 on average, and this is lower than in the C\(_2\)S and C\(_3\)S phases. Therefore, Ca(OH)\(_2\) precipitation (as portlandite) is always being in hydration process [12-13]. At the first hours of hydration process, few amounts of C-S-H phases and portlandite are formed. The amounts of C-S-H and portlandite increase with time, and C-S-H phases became the main hydration products after nearly 24 hours. In the beginning of hydration the dissolution of the clinker phases is partly prevented by initial hydrates which formed around the clinker grains. It was also expressed that the dissolution of C\(_2\)S is slower than the dissolution of C\(_3\)S [11-16].
There are several aluminate hydration products due to different concentrations of ions available in the pore solution. Hydration reactions of aluminate and water can be described as:

$$2\text{Ca}_3\text{Al}_2\text{O}_6 + 21\text{H}_2\text{O} \leftrightarrow \text{Ca}_4[(\text{OH})_6]_2(\text{OH})_2\cdot 6\text{H}_2\text{O} + \text{Ca}_2[(\text{OH})_4]_2(\text{OH})_2\cdot 3\text{H}_2\text{O}$$

which react to

$$\text{Ca}_4[(\text{OH})_6]_2(\text{OH})_2\cdot 6\text{H}_2\text{O} + \text{Ca}_2[(\text{OH})_4]_2(\text{OH})_2\cdot 3\text{H}_2\text{O} \leftrightarrow 2\text{Ca}_3[(\text{OH})_6]_2 + 9\text{H}_2\text{O}$$

The reaction of pure C₃A with water is very rapid and would lead to flash setting, which is prevented by the addition of gypsum to the cement clinker. If there is enough sulfate content, aluminate reacts to form ettringite

$$\text{Ca}_3\text{Al}_2\text{O}_6 + 3(\text{CaSO}_4\cdot 2\text{H}_2\text{O}) + 26\text{H}_2\text{O} \leftrightarrow \text{Ca}_6[(\text{OH})_6]_2(\text{SO}_4)_3\cdot 26\text{H}_2\text{O}$$

or to form monosulfate

$$\text{Ca}_3\text{Al}_2\text{O}_6 + \text{CaSO}_4\cdot 2\text{H}_2\text{O} + 10\text{H}_2\text{O} \leftrightarrow \text{Ca}_4[(\text{OH})_6]_2(\text{SO}_4)\cdot 6\text{H}_2\text{O}$$

The precipitation of ettringite and/or monosulfate leads to a continuous removal of the sulfate from pore solution. Therefore, the calcium sulfates dissolve slowly and are consumed within 8 to 16 hours period.

It is proposed that the hydration of calcium aluminate ferrite yields similar hydration products as those formed from aluminate: either iron is partly incorporated into the hydrates or precipitates as a Fe-(Ca-)rich gel.

It has been discussed that setting is not directly related with C₃A concentration. However, both setting and strength development is largely caused by hydration of C₃S to tobermorite (which is a differential composition of C-S-H phase) [17].

### 2.3 Cement Grinding Process

Grinding process is the comminution of materials to powder form. Clinker, produced in rotary kilns, has to be ground with the addition of gypsum to get the finish product, cement. The objective of the final grinding operation is to increase the specific surface of cement components – with a proper particle size distribution –, and to provide convenient reactivity of cement when used in concrete [18].
2.3.1 Cement Grinding Technologies

2.3.1.1 Ball Mill

Most common and extensively used cement grinding system is the ball mill technology which is based on rotating the grinding media and the material in a horizontal cylindrical shell. The grinding process applied by grinding media and liners inside the shell. Material and media lift at one side while the mill body is rotating, and fall down after reaching a height. That height is related with mill rotational speed, type of the liners, grinding media characteristics, filling ratio of the mill and properties of material [6].

There are two compartments divided by a diaphragm. First compartment is 30-33% of the overall length, and lifting liners with a ball charge from 50 mm to 90 mm are employed for size reduction. Then materials pass into the second compartment when the sizes are less than 2-3 mm, and grinding operation is applied with 15 to 50 mm sized balls or cyleps in the second compartment [17].

Cascading and cataracting are the main grinding actions in the ball mill process. When lifted media fall on to the particles, impact and percussion forces reduce the sizes, and that action is known as cataracting. Another motion is the cascading which grinds particles by flowing and rolling rather than falling. Compression and shear forces are effective at cascading motion. Those motions are used at different zones in the mill. Cataracting is more effective at first compartment where the coarser particles are broken, and cascading is used at second compartment to pulverize particles [18].

Ball mills normally operate about 75% of critical speed (the speed at which centrifugal force will just hold charge to the shell during rotation), and 25-35% volumetric charge loading. Circulating load is generally 200-300% with mechanical separators, and 150-200% with high efficiency separators.

However, the energy efficiency of ball mills is very low, especially for coarse grinding. Friction between the particles, grinding elements and liners is converted into heat, noise and electrostatic charge. Moreover, elastic and plastic deformations of materials and elements, and formation of particle agglomerations also cause losses [19].
2.3.1.2 Roller Press

In recent years, new grinding processes have started to be used as an alternative to the existing technology, the ball mill. One of the most commons is the roller press (also defined as high pressure grinding rollers (HPGR) in literature), which is also a relatively new comminution device that offers less energy requirements and improved capacities [20]. Comminution in the roller press is the result of high interparticle stresses which are generated by compressing a bed of solids between two pressurized rolls. Finer particle amount after that interparticle stress is much higher than conventional breaking and crushing techniques [21].

Roller press breaks particles in an autogenous way, unlike other conventional ball mill technology. The grinding force is transferred from one particle to other one, with only small proportions of the particles coming into direct contact with the rollers [20]. Although it is determined that interparticle process has less efficiency than single particle stressing, compressing a bed of particles and reducing the sizes by that force is more effective than ball mill. The reason of that conflict is the higher proportion of available energy being used exclusively for the purpose of stressing the bed of materials. However, stressing of materials inside the ball mill occurs randomly because of hit-and-miss nature of the process. Accordingly, unproductive collisions between balls and liners cause waste of energy [20-21].

Roller press has a simple design and working principle. One of the rollers rotates on a fixed axis while the other roller is allowed to move linearly towards or backwards from the fixed one according to the applied pressure and pressed material dispersion. The moveable roller is forced to press materials, which is placed in the gap between two rollers, by a hydraulic oil cylinder system [20]. The compressed materials formed a cake and agglomerated after passing between the rollers. Accordingly, disagglomeration is applied by generally V-separator system which is mainly a subsequent classifier. It has a static configuration of stepped plates down which the materials cascades through a cross flow of air [17].
2.3.1.3 Combined (Hybrid) Grinding System

General usage of roller press in cement grinding operations is as a pre-grinding unit prior to conventional ball mill system. Accordingly, efficient energy usage of roller press and grinding ability of ball mill are combined to get high reduction ratios with higher capacities. Roller press weakens the particles by compression, and microcracks are formed on the particles. Therefore Bond Index values of materials are reduced after passing through the roller press, and less effort required at ball mill stage to pulverize the materials. Increasing feeding rates and dropped specific consumptions result 10-50% energy savings when compared with closed circuit ball mill operations [23].

With a pre-grinding operation, large ball charge can be replaced with 20-25 mm balls at mill, and mill capacity is increased by 20%. The most common circuits are pregrinding with slab recirculation and semi-finish grinding [17].

2.3.1.4 Horizontal Roller Mill (HOROMILL®)

With the development of roller presses, pre-grinding applications have become an alternative for decreasing power consumptions. However, ball mill with roller press system include belt conveyors, hydraulic systems, elevators, weighfeeders, gas channels and classifiers with a ball mill and a roller press. Because of these several units and parts, process difficulties are observed and efficient process conditions could not be reached properly. According to that view, new designs have been investigated to improve energy efficiencies and system layouts, and HOROMILL® produced by FCB as a result of that research study [24].

After the tests on that pilot HOROMILL®, in September 1993, the first HOROMILL® began to produce cement in Trino, Italy. Although some technical problems were observed; the grinding principle of the first HOROMILL was effective, and the results were positive [25].

Although energy efficiency is the main advantage of this system, one of the other advantages is the structure of HOROMILL®, which is small and compact when compared with other grinding systems. Moreover, production flexibility is an advantage of HOROMILL®. The type of the cement can be changed automatically without stopping the plant. The control system changes the set points, and ratios of feeding materials get changed. The type of the product can be changed within 10 minutes [25].
Construction, feasibility and cost parameters were considered and the simplest design was selected in the production of HOROMILL®. As seen from Figure 2.1, the basic configuration is based on one idle roller within a cylindrical shell. The shell is driven in rotation by a gear motor with the help of a rim gear and a pinion. The grinding force is transmitted to roller by hydraulic cylinders. Internals are provided to control the material recirculation.

It was illustrated in Figure 2.2, the material advances regularly inside the mill with the help of centrifugal force and internal parts. The hydraulic roller exerts a pressure on the grinding bed, often causing material to become attached to the inner face of the shell by hypercritical shell speed. Scrapers are employed to remove this material, which then falls onto a diverting system, which pushes the material against the shell face for regrinding and adjusts the motion of this material inside the mill. The material is ground several times before leaving the mill, which permits an important comminution work while operating at moderate pressure about four or five times lower than in the roller presses.
Angles of nip, in Figure 2.3, which is the contacts of concave and convex geometries of the grinding surfaces, is two to three times higher than the angles in the roll presses, and also 1.5 times higher than the angles in the vertical roller mills. Higher angle of nip leads to thicker ground layer and more effective grinding work [24].
2.3.2 Classification Systems

Air separators have been used to classify the cement particles into two streams. Even distribution of particles is important at subjecting to classifying forces inside the separator. Centrifugal force, drag force and gravity are the main forces at classification, and efficiency of the process is based on these forces [6]. If the force of air flow on the particle is higher than the resultant of centrifugal force and gravity, the particle is carried with the air flow. If the amount gravitational force over particle is higher than the other active forces, then the particle falls down, and if centrifugal force defeats the other forces on the particle, particle is precipitated after accumulated at side walls [18].

2.3.2.1 First Generation Separators

Former separation technologies are known as the mechanical air separators, and these are first generation separators. A distribution plate is used to disperse the feed into the separating zone. This type of separators generates the circulation air inside the separator itself. The distribution plate throws the particles, then the heavier particles settle by gravity and some particles also hit the separator wall. Cyclone type wall also forces the particles down as tailings. On the other hand, finer particles lifted by air flow and transferred to fines cone. Air vanes are placed at the separating zone, and the separation is performed by to change the direction of flow and to decrease the velocity of air stream [17]. Small particles are always suspended in the air current because of low rate of descent, and a portion of fine particles is continuously circulate inside the system due to separation principle of the design [6].

2.3.2.2 Second Generation Separators

First generation separators suffered from high by-pass and difficulty in changing the particle size distribution and surface area of the fine product. Then, second generation separators were designed with main features of an external fan to circulate the air which replaced the internal fan, several external planetary cyclones which replaced the fines cone, independent control of separator speed and air circulation.
The air containing the fines is sent to the cyclones where the solids are collected from air stream, and then recycled the air stream back to the separator by fan system. The separation is based on the same principle as the first generation separators. The main advantages of the second generation separators are better removal of fines, sharper separation, lower by-pass and continuous control of the fineness. The main disadvantage is the size of the system. Dispersion of feed is still not enough and fine particle recirculation is not solved completely.

2.3.2.3 Third Generation Separators

Higher efficiencies required and separator systems were improved, then third generation separators were designed. The improvements with that system are;

- Uniform air velocity with horizontally entrance of air flow,
- Dispersion of feeding particles are more effective with changing the location of the distribution plate to the top of the air flow,
- A rotating cage is used to improve the selectivity of the finer and coarser particles,
- Separating size is adjusted by changing the rotor speed of the cage system,
- Air stream from the mill is used without any fine particle recirculation phenomena, because the fine particles are removed before recycling.

In operation, the air passes through the stationary guide vanes and the feed material is dispersed in the annular gap between these and the rotor. After passing through the vanes the air moves in a horizontal vortex. The air carries the fine material tangentially across the face of the rotor that is turning in the same direction as the vortex. The coarse particles are separated by a combination of gravity, centrifugal and impact forces and fall into the collecting cone at the base. The fine particles are conveyed to a dust collector. The sharp classification and low by-pass reduced the circulating load in tube mill-separator circuits and allowed an increase in feed rate by 20-40%. The specific energy consumption was reduced by 15-35% [6].
2.3 Effects of Grinding Techniques on Specific Energy Consumptions

Genc and Benzer [26] investigated the performance of different finish cement grinding circuits with high pressure grinding rollers. Capacity improvement and specific energy savings are observed with HPGR units before ball mill grinding systems. It was suggested that open circuit and hybrid HPGR applications increase grinding capacities with slightly less energy consumptions. However, obviously higher specific energy advantages are realized with closed circuit HPGR applications. Disagglomeration is stated as critical step in closed circuit HPGR applications. Agglomerated particles cause less efficient classification in the separator and higher circulating load. It is discussed that HPGR addition before an existing ball mill system change the feed size of the ball mill circuit. Accordingly, sizes of ball mill compartments, grinding media charges, design of the diaphragm and the operating parameters of the separator have to be optimized in order to get best results with an HPGR unit.

Genc and Benzer [26] also compared the size reduction efficiencies and specific energy consumptions of HOROMILL® and two-compartment ball mill with HPGR by operational data of the circuits. High additive contained Portland Puzzolanic cement is used at representative productions of study. It is suggested that when the operational parameters and size reduction capabilities of HOROMILL® and Hybrid grinding system (HPGR/two-compartment ball milling) are investigated, higher strength performance could be taken by producing Portland Puzzolanic cement with HOROMILL®. Especially, classification performance of HOROMILL® is stated as better than tested hybrid system. The specific energy consumption figures also showed that approximately 15% energy is saved with HOROMILL® usage.

Fuerstenau [27] presented there is an optimum range for dividing grinding energy between the roller press and the ball mill in a hybrid grinding system. Nearly 50-60% of the total energy used in roller press depending on the desired product size. It was cited that higher efficiency of hybrid grinding systems are based on two issues. Firstly, particle-bed comminution system of roller press is increased the efficiency of energy usage. Secondly, damaged and weakened particles are ground easily at ball milling stage after roller press system. This is evidenced by the increased breakage rates of roller press products relative to particles without pre-comminuted by roller press.

Tavares [22] explained that comminution in the roller press and subsequent ball milling generates significant energy savings when compared with single ball
milling operation. Compression of the bed of particles causes weakening results. It was studied to investigate weakening effectiveness of roller press systems according to the particle size, specific energy input and particle position within the bed. It was found that coarse particles are weakened more significantly with higher pressures and weakening is higher than any other conventional crushing equipment. It was also suggested that the energy saving from roller press systems is a combined result of the weakening of the particles and higher proportion of fines is generated in the product of the HPGR in comparison to other crushing methods.

2.4 Effects of Particle Size Distributions and Microstructural Properties on the Performance of Cement

Size, distribution and composition of Portland cement particles have a great effect on hydration kinetics, microstructure development and final properties of cement-based materials. Bentz et al. [28] inspected the effects of cement particle size distribution on a variety of performance properties which were studied through computer simulation and experiments. The effects of cement particle size distribution on performance properties of resultant products are stated as:

- Longer setting time is required with coarser cements, although setting achievement occurred with a lower heat release at hydration. Furthermore, strength developments of coarser cements are lower than finer ones.
- Hydration temperature releases are low for coarser cements, and consequently, coarser cements have less thermal cracking potential.
- More hydration is required for coarser cements to depercolate the capillary porosity, and improved curing could be possible.
- The diffusion coefficients for the coarser cements are much higher than those for the finer cements. At the same degrees of hydration, diffusivity of coarser cements is two times higher than finer cements.
- Properly cured coarser cement compositions create less empty porosity formations.
It is suggested that if improper curing conditions are possible, then finer cements are proper to use because of their increased early hydration rate and minimized loss of water with earlier de-percolation of the capillary porosity. However, it is cited that there is not a fixed ideal particle size distribution for cement; it has to be optimized for each different application [28].

Celik [29] stated that particle size distribution, uniformity of the distribution and specific surface area have a definite influence on service properties of cement, especially on strength. It was studied with samples of $-10 \, \mu m$, $-20 \, \mu m$, $-30 \, \mu m$, $-45 \, \mu m$, $-32+3 \, \mu m$ and $-20+5 \, \mu m$ fractions from PC 42.5 R type of cement by using a high efficiency laboratory separating system. Experimental studies supported that finer particles have a great effect on 2-day strength, but coarser particles are more effective at later ages (Figure 2.6-2.7). When $-32+3 \, \mu m$ fraction in the PC 42.5 R sample was increased by 20% (Figure 2.4), 28 days strength values were increased by 13%. On the other hand, addition of the $-20+5 \, \mu m$ fraction improved the early strengths, and a steeper size distribution with lower Blaine specific surface area was obtained (Figure 2.5). However, 28 days strengths were increased by 5%. It was also suggested that fine and coarse limits of the distribution have varying effects on strength development, and higher strengths could be obtained by more uniform size distributions with eliminating the tailing sizes (both fine and coarse tailing sizes).

Figure 2.4: Laser particle size distributions of PC 42.5 R samples including varying addition amounts of $-32+3 \, \mu m$ fraction [29]
Figure 2.5: Specific surface area values of PC 42.5 R samples with additions of varying $-32+3 \, \mu m$ and $-20+5 \, \mu m$ fractions [29]

Figure 2.6: Compressive strength values of PC 42.5 R sample and different size fractions of that sample [29]
Aiqin et al. [30] studied the influence of particle size distribution on cement properties, and suggested that packing density and hydration degree are the mainly affected properties. It was introduced that packing density is increased by wider particle size distribution, and homogeneity of the distribution increases the degree of hydration. Moreover, it was proposed that packing density is more important than hydration degree at early ages; however both packing density and hydration degree are not discarded for later ages. Accordingly suitable size distribution has to be generated for best results at later strengths, but wider size distributions are more successful at early strengths.

Celik and Oner [31] studied better reactivity of clinker mineral phases due to intergranular breakage along the grain boundaries at HPGR when compared to ball mill grinding operation, and effects on hydration properties of cement.
As illustrated at Figure 2.8, Blaine and BET specific surface area measurements were applied to both products of ball mill and roller press systems. Better packing ability of ball mill particles (because of friction based grinding principle) caused small voidages. Accordingly, high resistance to internal air flow occurred at Blaine measurements and specific surface area results of ball mill products were clearly higher than roller press product. However, when the other method, BET, was applied with same samples, specific surface area of the roller press particles are higher than ball mill particles. That situation was explained with the irregular surface formation and microcracks of roller press particles due to high compression pressure at size reduction. That surface structure conditions were supported by visual microscopic investigations.

Celik and Oner [31] studied the mineralogical composition of clinker particles in the roller press and ball mill products. It was explained that the clinker particles are composed of interstitial clinker phases, especially belite spots are placed inside of the alite grains at finer sizes. In the roller press, fractures occurred along the boundary zones of grains with high compression forces on particles, and uniformly liberated phases are obtained (Figure 2.9). However, at ball mill, transgranular fractions (fractured from the edges or weak points rather than fractures along the boundaries) occurred with repeating collisions of steel balls. That situation is also supported by measuring the amount of belite spots inside the alite crystals at fine sizes in Table 2.3.
During the hydration process, water contacts the surfaces and penetrates microcracks of particles, and it was expected that better liberated particles provide advantages in the hydration process.

Table 2.3: Amount of belite spots embedded in alite crystals [31]

<table>
<thead>
<tr>
<th>Size fraction μm</th>
<th>HPGR</th>
<th>Ball mill</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>$C_3S$ area (%)</td>
<td>$C_2S$ spots area (%)</td>
</tr>
<tr>
<td>$-63+53$</td>
<td>95.29</td>
<td>4.71</td>
</tr>
<tr>
<td>$-45+38$</td>
<td>97.16</td>
<td>2.84</td>
</tr>
<tr>
<td>$-32+20$</td>
<td>97.25</td>
<td>2.75</td>
</tr>
</tbody>
</table>

Figure 2.9: Liberation degree of Alite Crystals [31]
3.1 Materials Used in the Study

At the beginning, the main target of experiments was to obtain discussible results from stable industrial grinding process. However, the conditions had to be unique to obtain comparable data. Accordingly, producing CEM I 42,5 (Ordinary Portland Cement) type cement was selected as the experimental product. Main components of this type of cement are clinker and gypsum, but also limestone or any other component could be added as minor component without exceeding 5% (EN 197-1 Cement Producing Standards) of total mixture. Limestone is used as a minor additive in the plant where this thesis study was undertaken. However, for the purpose of this study, limestone was not added because Blaine fineness is affected significantly by the presence of limestone due to its easier grindability. Therefore, 95% clinker and 5% gypsum mixture was used to produce CEM I 42.5 in COMFLEX® grinding (roller press+ball mil) and HOROMILL® grinding systems.

There are individual bins for each component in the plant. Automatically-controlled feeding units set the speed of the belt conveyor under the bins according to the sectional weight over the conveyor. Operator inputs the total hourly feed rate and individual ratios of the cement components to automation system. Then, the total feed rate is distributed to each component according to the inputted ratios, and each component fed to the system by weighfeeders.

3.2 Raw Materials

3.2.1 Clinker

For the production of cements, the Ordinary Portland Cement (OPC) clinker of Denizli Cement Plant was used. Productions of cements were carried out with the same clinker lot to eliminate the effects of variations in clinker quality. Moreover, clinker of the same quality had been taken to the bins of each grinding system. Chemical analyses of the clinker are given in Table 3.1. Main clinker phases were also calculated according to Bogue’s Equations and given in Table 3.2. Shape
and distribution of crystal formations were inspected with microscopic analyses which are shown in Appendix H. According to SEM photographs, although there were some heterogeneous characteristics, formation was generally proper to produce cement.

### Table 3.1: Chemical and Physical Analyses of Ordinary Portland Cement Clinker Samples

<table>
<thead>
<tr>
<th>Loss on ignition (%)</th>
<th>SiO₂ (%)</th>
<th>Al₂O₃ (%)</th>
<th>Fe₂O₃ (%)</th>
<th>CaO (%)</th>
<th>MgO (%)</th>
<th>SO₃ (%)</th>
<th>Na₂O (%)</th>
<th>K₂O (%)</th>
<th>Alkali Equivalent</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.30</td>
<td>21.07</td>
<td>5.17</td>
<td>3.88</td>
<td>65.47</td>
<td>1.77</td>
<td>1.43</td>
<td>0.14</td>
<td>0.77</td>
<td>0.65</td>
</tr>
</tbody>
</table>

### Table 3.2: Main Clinker Phases and Other Components of the Test Samples

<table>
<thead>
<tr>
<th>Lime Saturation Factor</th>
<th>Modulus of Silica</th>
<th>Modulus of Aluminum (A/F)</th>
<th>C₃S (%)</th>
<th>C₂S (%)</th>
<th>C₃A (%)</th>
<th>C₄AF (%)</th>
<th>Liquid Phase (%)</th>
<th>Cl (%)</th>
<th>Free CaO (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>96.82</td>
<td>2.33</td>
<td>1.33</td>
<td>55.44</td>
<td>18.61</td>
<td>7.14</td>
<td>11.81</td>
<td>26.92</td>
<td>0.0024</td>
<td>1.61</td>
</tr>
</tbody>
</table>

#### 3.2.2 Gypsum

Like OPC clinker, gypsum of Denizli Cement Plant was used in the plant experimental study. Gypsum has been purchased from a local quarry which is nearly 12 km away from the plant. Chemical analysis of gypsum is given in Table 3.3.

### Table 3.3: Composition of Gypsum

<table>
<thead>
<tr>
<th>Moisture Content (%)</th>
<th>SO₃ (%)</th>
<th>Relative Water (%)</th>
<th>SiO₂ (%)</th>
<th>Al₂O₃ (%)</th>
<th>Fe₂O₃ (%)</th>
<th>CaO (%)</th>
<th>MgO (%)</th>
<th>Other (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>0.92</td>
<td>43.93</td>
<td>18.43</td>
<td>0.29</td>
<td>0.01</td>
<td>0.03</td>
<td>33.56</td>
<td>0.57</td>
<td>2.26</td>
</tr>
</tbody>
</table>

#### 3.2.3 Other Materials

IDEA PSI-502 grinding chemical has been used in the plant for sustaining the grinding performance and later strength, and this practice was not changed during the study. All laboratory materials like water, standard testing sand as
defined in TS EN 196-1 and other material requirements were obtained from Denizli Cement Plant for the preparation of mortars and prisms.

### 3.3 Testing Equipment and Standards

The chemical and physical analysis of clinker and cement samples were conducted in the chemistry and physics laboratories of Denizli Cement Plant. Strength, setting time and other relevant product based analyses were also done in the concrete laboratory of the plant. The list of equipment and analyses used in the study are shown in Table 3.4 together with the relevant standards.

#### Table 3.4: Cement Tests, Equipment and Related Standards

<table>
<thead>
<tr>
<th>Type of Analysis</th>
<th>Testing Equipment</th>
<th>Referenced TS EN Standard</th>
</tr>
</thead>
<tbody>
<tr>
<td>Determination of Sieve Residue</td>
<td>Alpine Jet Air Sieve</td>
<td>TS EN 196-6 Methods of Testing Cement Part 6: Determination of fineness</td>
</tr>
<tr>
<td>Determination of Consistency</td>
<td>Vicat Apparatus</td>
<td>TS EN 196-3 Methods of Testing Cement Part 3: Determination of Setting Time and Soundness</td>
</tr>
<tr>
<td>(Water Demand)</td>
<td></td>
<td></td>
</tr>
<tr>
<td>Determination of Soundness</td>
<td>Le Chatelier Mould</td>
<td>TS EN 196-3 Methods of Testing Cement Part 3: Determination of Setting Time and Soundness</td>
</tr>
<tr>
<td>Determination of Density</td>
<td>Le Chatelier Flask</td>
<td>TS EN 196-6 Methods of Testing Cement Part 6: Determination of fineness</td>
</tr>
<tr>
<td>Determination of Fineness</td>
<td>Blaine Apparatus</td>
<td>TS EN 196-6 Methods of Testing Cement Part 6: Determination of fineness</td>
</tr>
<tr>
<td>Determination of Cement Strength</td>
<td>Mortar Mixer Jolting Table Cement Moulds Compression Machine</td>
<td>TS EN 196-1 Methods of Testing Cement Part 1: Determination of Strength</td>
</tr>
</tbody>
</table>

Particle size scanning electron microscopy analyses were performed in the laboratories of the Turkish Cement Manufacturers Association.
3.4 Cement Grinding Systems

3.4.1 COMFLEX® Grinding System

In COMFLEX® grinding system, (Figure G.1-3; J.1; I.1 in Appendix G-J-I) material mixture is fed directly to two roller presses. Both rolls have individual drive units which are synchronized to each other. One roll of the roller press is fixed in position and rotates with the force of the drive system. The other roll is the moving one, and the pressure system is placed on it. There is a gap between the rolls which is measured by sensors. When the material is fed between the rolls, it is drawn into the compression zone between the rolls while the compressed material forces the rolls apart. If the gap between the rolls starts to increase, the hydraulic pressure unit works and pushes the moveable roll, and presses the material to maintain the gap. The magnitude of the applied pressure is controlled by an automatic valve system which works according to input parameters such as pressure and gap settings. The adjustable pressure makes the system more flexible in equipment performance, energy consumption, and product quality requirements.

The material crushed and fractured in the roller press is dropped into the V-separator. The V-separator, as the name implies, is a V-shaped static air separator without any moving parts and there are built-in step grates inside of it. Material is fed from the middle and falling particles are affected from the air flow between the step grates. Fine particles are carried upwards with the air through the separating channels, and the coarser ones fall down over the step grates and are discharged at the bottom of the separator housing. With such a simple separating system coarser particles are separated before an exact classification and the agglomerated crushed particles of the HPGR are disagglomerated, and, hence, the separating efficiency of the system is increased. Also, the drying application could be undertaken by the V-separator with hot gas if moisture contents are high.

The coarse product of the V-separator is recirculated back into the roller press. The fine material is transported by air and goes to a third-generation dynamic separator having a rotational cage system inside. The dynamic separator is much more efficient than the V-separator and from any other type of static separator as well.

The fine product stream of the dynamic separator is collected in the bag filter system. The fine particles collected over the filter fabric constitutes the cement product which is dispatched into the cement silo.
Coarser particles, which are collected at the bottom of the dynamic separator, flow into a small bin. There are two outlets of the bin, one at the bottom and one at the top. There is an adjustable flap on the air slide of the bottom outlet. If the flap is closed or the bottom outlet flow is restricted, then the material content inside the bin increases, and eventually the material overflows from the second outlet at the top. In other words, coarser particles from the dynamic separator are divided into two parts by a flow-controlled bin system. The bottom outlet flow is directed to the roller press recirculation and the top outlet flow goes to the ball mill system. By this way, the ball mill rate is controlled according to mill conditions (load of the mill system is checked from the motor current of the bucket elevator of the mill system).

It can be seen that some particles are only pressed by the rollers and do not pass through the ball mill because the required fineness has been reached at the roller press unit. However, some material is sent to the ball mill after being crushed in the roller press. This process yields a cement product which is composed of material with mixed physical characteristics which may affect concrete properties.

3.4.2 Roller Press System

Although COMFLEX® Grinding System is composed of roller press and ball mill units; the roller press unit is also operated in the finish grinding mode producing cement individually (Figure G.5-6; I.3; J.3 at Appendix G-I-J). As explained in the previous section, cement is produced partly by the roller press and partly by the roller press-ball mill line in the operation of COMFLEX® Grinding System. There is a dividing gate under the dynamic separator of the roller press unit, and the coarser material stream is divided into the ball mill feeding line and the roller press return line. When the ball mill is not operated, the coarse material is recirculated only inside the roller press line and continued producing cement with 110-120 tph capacity. This alternative grinding process is also investigated and analyzed in this study.

Arrangement of this grinding process is similar to the COMFLEX® system till the dynamic separator. Feed material is transported into the roller press bin with belt conveyors and bucket elevator. Then, the material is fed over the rollers, and crushed material drops into the V-separator. Primary classification is done in the V-separator, and the finer particles are carried with air stream to the dynamic separator. Sharper and efficient classification is achieved in the dynamic separator, and finer particles are sent to silo after being collected in the bag.
filter. However, the coarser particles of the dynamic separator are recirculated to the roller press bin, mixed with the fresh feed, and fed to the rollers for grinding.

Simple crushing and breaking process, efficient energy consumption and rapid response to operational tunings are the main advantages of this alternative process. Nevertheless, produced cement particles have microcracks and irregular shapes, which caused various adverse effects in concrete production stages, such as higher porosity, increasing water demands, change in the workability.

### 3.4.3 HOROMILL® Grinding System

The HOROMILL® (horizontal roller mill) is made up of a horizontal shell equipped with a grinding track within which a roller exerts the grinding force. The shell is rotated by a gear attached to its end. The mill is supported by four hydrodynamic shoes ensuring perfect stability. The roller going through the shell is rotated by the material. No drive is necessary, hydraulic pressure and contact with material cake enables the rotation of the roller.

The pressure transmitted to the roller by two traction jacks located outside the mill and secured to the articulated arms. The pressure which is necessary for the operation of jacks in the working phase is generated by a hydraulic pressure unit. The hydraulic system is also used in maintenance phases.

A lubrication unit ensures an efficient lubrication of the shoes and driving gear. System stability has to be controlled accurately due to high working pressures. Moreover the system uses the same lubrication system and the same oil for the hydrodynamic shoes and the driving gear.

The driving gear of the mill consists of a gear and a pinion. Pinion adjustment is made by means of eccentric gears and wedging.

The ends of the mill consists of two heads to which are connected the feeding inlet, dedusting pipe and material discharge devices, the material advance system and the scrapers.

Operating principle of HOROMILL® is basically based on the rotating shell of the mill rotated faster than the critical speed, which results in centrifuging the material introduced into the mill. The mill is fed through an opening at the upper part of the feed head; the material falls by gravity into the first low part of the mill to be centrifuged. Then scraping tools - provided in the upper part of the shell and covering the entire length of the mill - scrape off the material which
falls onto the adjustable plate of the material advance system. The material advance system (material forward) is so oriented (sloping towards the discharge end) as to enable the product to advance. Depending on its position, it is possible to make the material advance slower or faster in the mill and hence to adjust the number of passages under the roller. That material forward is operated and positioned by a servomotor (controlled from the automation room). When the material has gone through the length of the roller, it is conveyed toward the discharge chute, and the ground material leaves the mill. Then, classification of particles is applied at a 3rd generation separator, and coarser particles turn back to system for re-grinding until reaching the desired fineness. On the other hand, finer particles are transported with air to the bag filters and collected in the filter system as final product. (Figure G.4; I.2; J.2 at Appendix G-I-J)

3.5 Experimental Program

The experimental program of this study is composed of six stages:

i. Determination of chemical, physical and other specific characteristics of raw materials according to the TS EN standards.

ii. Preparation of grinding systems and operating the systems with targeted fineness value of 3600 cm²/g of products.

iii. Application of sampling process after getting steady state operations of the three different grinding systems.

iv. Determination of particle size distributions and particle shape properties of the samples.

v. Determination of physical, chemical and hardened mortar properties of products:
   a. Sieve residue and Blaine fineness tests according to TS EN
   b. Chemical composition tests with XRF analysis of products
   c. Specific gravity, setting time, compressive strength, soundness and water demand tests according to TS EN standards

vi. Determination of grinding efficiencies of operational observations, separator efficiencies and specific energy consumptions.
3.6  Industrial Operations and Sampling Periods

3.6.1  COMFLEX® Grinding System (Roller Press + Ball Mill)

Roller Press and Ball Mill system was operated according to the decided feeding proportions of 95% clinker and 5% gypsum. Before sampling, the system was run until reaching stable operating conditions. Then, the sampling operation was started while the system is operating, and Blaine values of the final product were measured to check if the target Blaine fineness of 3600 (±100) cm$^2$/g was achieved. Otherwise, speed of the separator and the system fan was adjusted accordingly and we waited nearly 30 minutes to obtain another set of reliable samples. After several trials, accepted ranges of Blaine fineness were achieved and 10 kg of sample was taken from each sampling point. The sampling operation was started and completed at the same time period for all sampling points in order to avoid time-dependent fluctuations in the grinding system.

3.6.1.1 Samples and Sampling Points (Figure G.1-3 at Appendix G):

(C.1) Finer particles of Roller Press system: Taken from 260AS02 airslide which was located under the 260BF01 main system filter of Roller Press unit.

(C.2) Finer particles of Ball Mill system: Taken from 260AS04 airslide that was located under the product collecting cyclone (260MC01) of the ball mill system.

(C.3) At the exit of the ball mill: Taken from 260AS01 airslide which was carrying the ground material to the classifying system of the ball mill unit.

(C.4) Return of the ball mill: Taken from 260AS03 airslide which was sending the coarser particles of classifier to ball mill again.

(C.5) Return of the roller press: Taken from 260AS06 which was located under the dynamic separator of the roller press system, and sending material to ball mill system. Also some amount of the same material turns back to roller press.

(C.6) Mixed finer particles (product) of ball mill and roller press: Taken from sampling device on the airslide of the main product line. Both products of ball mill and roller press transported with individual airslide lines, but mixed in a main airslide line and sent to cement silos with a single line. By that way, different products mixed continuously before silo on a long transportation line (composed of a nearly 50 meters length of airslide system and bucket elevators).
There is a sampling point C.7 illustrated on Figure G.1 in Appendix G. That point was on the gas channel which transports the finer particles of V-separator to dynamic separator. Thus, that line was the feeding line of high-efficiency dynamic separator. However, representative sampling was not possible from that channel, and accordingly size distribution of that point was calculated with mass balance formulations (as given in Section 3.10.1). Mass flow rates were provided from solid flow meters (amount of flowing solids measured by sensors in the units of mass per time) on the airslide lines, and the size distribution was calculated by using this process data, and it was used at classification efficiency expressions (described in Section 3.10).

3.6.2 Roller Press System

Roller Press was operated with the same proportions of clinker (95%) and gypsum (5%) in the feed material as above. Sampling operation was started after reaching the steady-state operation of the roller press circuit. Fineness target was again 3600 (±100) cm²/g Blaine, and the representative samples were taken after checking the fineness value as described above.

3.6.2.1 Samples and Sampling Points (Figure G.5-6 at Appendix G)

(R.1) Finer particles of Separator (Product): Taken from 260AS02 airslide which was located under the 260BF01 main system filter of Roller Press unit.

(R.2) Coarser particles of Separator (Return): Taken from 260AS06 which was located under the dynamic separator of roller press system, and sending material to ball mill system. Also some amount of the same material turns back to roller press.

Similar to sampling point C.7 in Section 3.6.1.1, size distribution and flow rate of sampling point R.3 (illustrated at Figure G.5 in Appendix G) was calculated with solid flow meters on the airslide lines.

3.6.3 HOROMILL® System

HOROMILL® system was also operated according to feeding proportions of 95% clinker and 5% gypsum. After achieving targeted Blaine fineness (3600 (±100) cm²/g), samples were taken with similar procedures applied in other systems.
3.6.3.1 Samples and Sampling Points (Figure G.4 at Appendix G)

(H.1) Finer particles of HOROMILL® system: Taken from the sampling device at product line which is located under the system filter and before the airslide line.

(H.2) Return particles of HOROMILL® separator: Taken from below of the separator. Those particles are coarser particles after separation process.

(H.3) Particles before the separator: Taken before the separator and composed of grinded material which is coming from the mill outlet.

3.7 Particle Size Distribution Analysis and Expression Methods

In order to investigate the grinding processes, ground materials had to be inspected on particle size basis. Accordingly, particle size analyses were performed with a laser diffraction particle size analyzer (PSA) to obtain particle size distribution. Not only the products of the systems but also other samples were analyzed.

During the laser diffraction measurement, particles are passed through a focused laser beam. These particles scatter light at an angle that is inversely proportional to their size. The angular intensity of the scattered light is then measured by a series of photosensitive detectors. The number and positioning of these detectors in the device have been optimized to achieve maximum resolution across a broad range of sizes. The map of scattering intensity versus angle is the primary source of information used to calculate the particle size. The scattering of particles is accurately predicted by models in software, and allowing accurate sizing across the widest possible dynamic range.

Characterizations of samples were obtained from the software of the analyzer. Categorization of amounts according to particle sizes and size distribution curves with histogram graphs were obtained. Furthermore, the size distributions were also plotted on specially prepared graph papers by using the Rosin-Rammler function, which is the commonly used particle size distribution model in the cement industry.
The Rosin-Rammler function is defined as

\[ Q_r(x) = 1 - \exp(-bx^n) \quad \text{or} \quad Q_r(x) = 1 - \exp\left(-\left(\frac{x}{x_e}\right)^n\right) \]  

where \( b \) is a constant equal to \( x_e^{-n} \), \( x_e \) is an absolute size constant and equal to \( x_{63.2} \), and \( n \) is the constant that shows the dispersion of particle sizes [32].

The same function for retained percentage is also rewritten as

\[ R = 100 e^{-\left(\frac{x}{k}\right)^n} \]  

where \( R \) is the volume of oversized material in percent, \( x \) is the particle size in mm, \( k \) is the absolute size constant and equal to 36.79, \( n \) is the size distribution constant [33].

### 3.8 Particle Shape Analysis

Particles of products from the three systems of this study were also inspected with the scanning electron microscope (SEM) photographic analysis.

The SEM images the sample surface by scanning it with a high-energy beam of electrons. The atoms of sample interact with the electrons. The energy exchanges, that caused by reflection and emission of electrons, between electron beam and the sample are detected by specialized detectors. Then surface topography, composition and other properties such as electrical conductivity of sample is obtained. As a result, the SEM provides high contrast images to identify the microstructural characteristics of cement particles.
3.9 Physical and Chemical Quality Analyses of Products

3.9.1 Sieving and Surface Area Analysis

During sampling periods, sieve analysis and Blaine fineness measurements were conducted. 45 µm residue analysis has been the control parameter of cement production in the plant. Accordingly, 45 µm residue analyses were also applied in the sampling period.

Sieve analysis was performed with Alpine air jet sieve to determine the amount of oversized particles in the samples. In sieving operations of very fine particles, blinding, that is, the obstruction of the sieve openings, affects the efficiency and accuracy of the operation [34]. Accordingly, the strong jet of air is used to prevent blinding by Alpine air jet sieves. Air jet increases the efficiency of sieving also by moving the particles without mechanical interventions like tapping or brushing. In the sieving operation, air flows upwards through a sieve from a controlled opening (rotating plate over the opening continuously changes the point of air flow), and maintains a fluidized state for materials. Another suction flow is applied to the bottom of the sieve to remove finer particles, and oversized particles retained over the sieve after a period of time.

Another physical analysis is determination of the specific surface area. Air permeability methods are generally used to measure the specific surface area of samples, and the Blaine method is the most common one in the cement industry. The principle of the method is drawing a fixed volume of air through a bed of powder of known porosity and density in a measured period of time. The pressure required to draw the air through the sample bed of powder is measured by the movement of oil in a monometer. The time required for the movement of oil to a stated level is recorded and used in the calculations [35].

All specific surface area measurements were performed by using the Blaine device in the laboratory of Denizli Cement in accordance with TS EN 196-6 standard. Firstly, density of a sample was measured by the gas pycnometer. This device works with pressurized helium gas at 1.5-1.7 bar. Density was obtained from the volume differentiation of gas after known volume of sample was added. Then, required sample quantity was found by the equation:

\[ m = d \times (1 - e) \times V \]  \hspace{1cm} (3)

where \( m \) is the required sample amount, \( d \) is the density of sample, \( e \) is the porosity of sample (taken as \( e = 0.5 \)), \( V \) is the bulk volume of sample.
Determined amount of sample was taken into the device with a filter paper to prevent the flow away of finer particles. Then air flowed through the sample and the time period of changing oil level measured by the Blaine device. At the end the time was substituted into the equation:

\[
S_w = K \times \left( \frac{e^3 \times t}{d \times (1 - e) \times P} \right)
\]  \hspace{1cm} \text{(4)}

Where \( K \) is obtained from the test of two representative samples which have known specific surface areas and densities, and those known values substituted into the below formula to get \( K \) constant:

\[
K = S_w \times \left( \frac{d \times (1 - e) \times P}{e^3 \times t} \right)
\]  \hspace{1cm} \text{(5)}

In equation (4), \( S_w \) is the specific surface area in units of \( \text{cm}^2/\text{g} \), \( K \) is the constant of device, \( t \) is the measured time interval, \( d \) is the density of sample \( (\text{g/cm}^3) \), \( P \) is the viscosity of air at testing air temperature (units of \( P \) is poise).

### 3.9.2 Compositional Analysis

Components of cement are also important in the quality case. Thus, ratios of main oxides and other contents have to be discussed to check the variances and to determine their possible effects on further tests. Conventionally, cement analysis was carried out using wet-chemical techniques. However, harder and time consuming techniques have been replaced by X-ray analysis equipment of various types. Nowadays, X-ray fluorescence (XRF) is used routinely in cement analysis because of providing rapid compositional data for controlling almost all stages of production.

The basic principle of XRF analysis is sending a beam of X-rays to cement sample. Then, X-ray beam causes the generation of X-rays within the cement, and also reflections occur. Those X-rays from the cement sample are collected by suitably placed X-ray detectors. The collected X-rays have various energy rates because they come from different atoms inside the cement sample. This means that each atom generates specific energy of X-rays because of their unique atomic number [36].
If these energies are measured under carefully-controlled conditions, the X-rays from each element could be counted over a period of time; the proportions of each element are obtained.

The specimen of cement or other material may be in the form of a glass bead or a pellet of pressed powder.

Beads are made by heating the specimen together with a flux, typically lithium tetraborate, at about 1100°C to form a glass. This approach has the advantage that the specimen is then a homogeneous material, allowing more accurate X-ray analysis.

Pressed pellets are made by grinding the specimen finely and compressing the resulting powder to form a pellet. This pellet is then analyzed directly. Preparing pressed pellets is quicker and easier than preparing glass beads, but the specimen is then a heterogeneous material. This makes the calculation of the processes of X-ray fluorescence and absorption within the specimen more complicated and there may be some loss of accuracy, although it should be minimal.

3.9.3 Mortar Analyses

Cement quality is evaluated with mortar tests because the main property of cement is supplying strength by holding together the components of concrete mix. Accordingly, cements are classified with their strength performances. Therefore, samples of the cement products from the tested operations had to be inspected on performance basis. For the determination of strength properties and also the other product characteristics, the concrete laboratory of Denizli Cement was used.

Strength tests were applied to mortars of the sampled cement products of each grinding system. These mortars were composed of cement, sand and water with standardized materials and procedures.

“TS EN 196-1:2009 Methods of Testing Cement / Part.1: Determination of Strength” is a standard about the materials, equipment and applications on the strength test of cement. Although produced cement was standardized with TS EN 197-1:2002 as defined previously, other materials and equipment had to be standardized for reliability and uniformity.
Thereby, testing of samples was done according to TS EN 196-1 standard [37]. CEN Standard Sand is one of the main components of strength analysis. CEN (European Committee of Standardization) sand is natural sand, which is silicon dioxide (min. 98%) based and cleaned. The particles are generally isometric and rounded in shape, and screened with defined size distributions. Particle size distribution of CEN sand has to be inside the ranges listed in Table 3.5.

Table 3.5: Size Distribution of CEN Standard Testing Sand [37]

<table>
<thead>
<tr>
<th>SQUARE MESH SIZE (mm)</th>
<th>2.00</th>
<th>1.60</th>
<th>1.00</th>
<th>0.50</th>
<th>0.16</th>
<th>0.08</th>
</tr>
</thead>
<tbody>
<tr>
<td>CUMULATIVE RETAINED BY WEIGHT (%)</td>
<td>0</td>
<td>7 ± 5</td>
<td>33 ± 5</td>
<td>67 ± 5</td>
<td>87 ± 5</td>
<td>99 ± 1</td>
</tr>
</tbody>
</table>

Besides cement and CEN sand, water is the other component to prepare test mortars. There is not an additional requirement for water but it has to be clean as drinking water according to the standard.

3.9.3.1 Preparation of Mortars and Prisms

As specified in the relevant standard; 1 unit of cement, 3 units of CEN sand and ½ unit of water were used to prepare test mortars. In order to prepare 3 test samples, 450 (±2) grams of cement, 1350 (±5) grams of CEN Standard sand and 225 (±1) grams of water were used.

Standard mixing procedure was applied to materials, and 40 mm x 40 mm x 160 mm sized moulds were used to produce mortar prisms. The prisms were cured inside 20(±1) °C temperature of water until required time period was reached.

1-day, 2-day, 7-day and 28-day strength measurements were taken to determine the performance of cement. TS EN 197-1 states cement classes and sub-classes for the strengths of 2, 7 and 28 days [38]. However, 1 or 3-day strengths also provide data about relations of early and later performances. Thus, 1-day strengths of samples were also measured in the study.

Moreover, curing periods are also limited in the standard as the following [37]:

- 1-day strength test has to be determined after 24 hours ± 15 minutes
- 2-days strength test has to be determined after 48 hours ± 30 minutes
- 7-days strength test has to be determined after 7 days ± 2 hours
- 28-days strength test has to be determined after 28 days ± 8 hours
Prisms were prepared for each sample to get 1, 2, 7 and 28 days strengths. Each prism was divided into two parts as explained in the standard, and strength tests were applied to both specimens. The arithmetic average of the results was taken as the strength value of that day.

All the equipment and measuring process was based on TS EN 196 standard like other preparation activities. Compression machine was used to test the pressure strengths of cement samples. The sample was located in the breaking zone which has a size of 40 mm x 40 mm like the prism. There were two unique samples because the prism was broken into two parts before testing (normally, samples are broken to two at the tensile strength test, but (in this study) two compressive strength test samples are aimed when the samples were broken). One part was placed in the breaking bed with a similar symmetrical position of bed and axis. Then the machine was activated.

The compression machine loaded the samples at a fixed rate of 2400 ±200 Newton/second until the samples were broken. The applied load achieved just before the breakage was divided by 1600 to get the Newton per square millimeter (N/mm²) or Megapascal (MPa) value. The strength performance of cements are defined and classified by N/mm² or MPa in the standards and literature [37].

\[
\sigma = \frac{P}{A}
\]  

(6)

where \( P \) is the average of load applied at breaking points in Newtons, and \( A \) is the cross sectional area of the application zone in units of mm².

### 3.10 Operational Efficiencies of Grinding Systems

#### 3.10.1 Classification Efficiency

Separator efficiency and Tromp curves were used to study the classification efficiencies of the grinding systems because the grinding system performance is directly related with classification stage. Misplacement of finer particles into the coarse product stream of the separator leads to capacity losses, and reduces the complete system efficiency.
Tromp curves were plotted using the PSA data, and the general equations of the closed circuit process are:

\[
A = F + R \tag{7}
\]

\[
(A \times a) = (F \times f) + (R \times r) \tag{8}
\]

\[
(A \times \Delta a) = (F \times \Delta f) + (R \times \Delta r) \tag{9}
\]

\[
U = \frac{(f - r)}{(a - r)} \tag{10}
\]

\[
\eta = \frac{f}{U \times a} \tag{11}
\]

where;

\(A, F,\) and \(R\) are the tonnage flow rates (tons/h) of the separator feed, separator fine product stream, and the separator coarse product stream, respectively. The coarse product stream is recirculated for regrinding.

\(a, f,\) and \(r\) are the percent of cumulative undersized weights of separator feed, fines and tailings at a defined size.

\(\Delta a, \Delta f,\) and \(\Delta r\) are the percent of differential weights in a defined size interval.

\(U\) is the circulating load percentage of the system.

\(\eta\) is the efficiency of the separator.

Tromp curves were plotted for each system to observe the sharpness of classifications. The Tromp curve shows the probability for a particle of a given size to escape with the rejects. The equation to calculate the coarse grade efficiency of separation, \(T\), for a given size interval is:

\[
T = \left[1 + \left(\frac{\Delta f \times \frac{1}{U}}{\Delta r \times \left(1 - \frac{1}{U}\right)}\right)\right]^{-1} \tag{12}
\]

where \(\Delta f\) and \(\Delta r\) are the weight percentages of the particles in a given size interval of the feed and the coarse product streams of the separator, respectively.
In the y-axis coarse grade efficiency that is calculated according to the Tromp efficiency equation is placed. Particle sizes are placed logarithmically in the x-axis of the graph. Sizes of 25, 50 and 75 percents are defined on the graph as $D_{50}$, $D_{75}$ and $D_{25}$, and showing classifying point and zones. Steepness of the curve expresses the efficiency of the separation.

3.10.2 Energy Efficiency

Energy consumptions of the systems during test production periods were also observed to compare the systems. There are main equipment and auxiliary units for grinding systems. Main equipments are the drive systems, system fans and separators. The transportation equipment like belt conveyors, airslides and elevators; and also weighfeeders, dust filters, hydraulic pressure unit with other specific motors in the systems compose the auxiliary units. Those secondary units have an integral counter of electric energy consumption that takes the values from motor control center (MCC) for each grinding system. However, energy consumptions of the main equipment were recorded by individual counters. As a result, average of hourly consumed kilowatt-hour was calculated from the counter values. Then, the specific energy consumption for each system in units of kWh per ton of cement was calculated.
CHAPTER 4

RESULTS AND DISCUSSION

4.1 Particle Shape Characteristics

Changes in the grinding mechanism affect the particle morphology and the performance of cement. Compression, shear and impact forces are applied on the cement particles during grinding stages. However, since the forces and acting zones differ between the grinding systems, shape and surface structures of particles are expected to be different, and such possible differences were determined with the SEM analyses which were executed in Turkish Cement Manufacturers Association (TCMA) laboratories as shown in Appendix F.

In the SEM images of the representative product samples, particle shapes and distributions were observed. Exact quantitative evaluations could not be made by a visual inspection of SEM images; however, it was seen that especially the roller press product particles and also some Horomill product particles had sharper edges and ball mill particles had more regular structures.

Celik and Oner [31] suggested that particles ground in the roller press had irregular rough surface structures, fissures and microcracks occurred due to high compression loads on the particles. They supported that suggestion with microscopic photographs and also comparative inspections of Blaine and BET specific surface analyses of samples from roller press and ball mill products.

According to the results of that study; Blaine specific surface areas for the same size ranges had different values when the grinding method was changed. Ball mill products had higher specific surface area according to Blaine tests. On the other hand, the same samples were tested with BET method, and HPGR products had higher specific surface area with that method. Then, it could be suggested that irregular surface formations and microcracks of HPGR products caused higher surface area results in BET analysis. On the other hand, rounded shaped ball mill particles with smoother surfaces formed a well packed mass which showed higher resistance to air flow in the Blaine specific surface area test, and gave higher surface area results. [31]
Similar results have been found when the plant operations were investigated. There is another two-compartment ball mill system (having no connection with roller press unit) which produces cement individually (if required). Rarely, the same type of cement (CEM I 42.5 R) has been produced with both the individual roller press system and the individual ball mill system. The specific surface area and Blaine fineness values of related production periods are illustrated in Figures 4.1 and 4.2.

**Figure 4.1:** Sieve residues of 15 samples from different grinding systems

**Figure 4.2:** Blaine fineness of sieved 15 samples in the same order
It was seen that specific surface area increases with finer grinding, and consequently Blaine values increase with decreasing amount of sieve residues (45µm is used as production parameter in the plant). However the ratio of changes was clearly different in roller press grinding when compared with ball milling. That difference was caused by surface structures and relative particle packing conditions.

Moreover, HPGR particles have rough and cracked surfaces due to high pressure. That force also caused dissimilar surface area formations and affects hydration reactions of cement particles in the former stages.

Belite (C$_2$S) grains embedded inside the alite (C$_3$S) crystals were inspected by Celik and Oner [31] to observe the differences of breaking and fracturing properties of particles in ball mill and high pressure grinding rollers (HPGR). Mineralogical analysis of -63+53 µm; -45+38 µm; -32+20 µm size fractions showed that alite contents increased with finer grind (Table 4.1). Moreover, C$_2$S and C$_3$S occurrence with finer grinding are seen from covering area proportions (Table 4.2). It is clear that C$_2$S contents decreased with finer grinding but there is also a variation between grinding methods. That is the reason of preferential microcracks and fractures along the boundary points of alite and embedded belite crystals because of high pressure on particles. However, repeated collisions of balls in the ball mill cause transgranular fractures (fractured at edges of lattices) rather than fracturing from boundaries of different grains. Therefore, with HPGR, fractured boundaries get broken easily and more effective surfaces for reactive crystals are obtained. In addition, at hydration stages, water easily penetrates through the fractures on particles. If fractures are on the boundaries, then alite and belite crystals are hydrated more effectively.

Table 4.1: Mineralogical compositions of main phases in size fractions of grinding modes [31]

<table>
<thead>
<tr>
<th>Size fractions (µm)</th>
<th>HPGR (%)</th>
<th>Ball mill (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td>Alite</td>
<td>Belite</td>
</tr>
<tr>
<td>-63+53</td>
<td>72.7</td>
<td>2.9</td>
</tr>
<tr>
<td>-45+38</td>
<td>77.9</td>
<td>4.5</td>
</tr>
<tr>
<td>-32+20</td>
<td>86.0</td>
<td>3.7</td>
</tr>
</tbody>
</table>
Same situations had also been observed at industrial stage when the operational data of the plant is observed. There has been another ball mill system at Denizli Cement Plant. That ball mill (Cement Mill-1) is completely similar with the ball mill of COMFLEX® Grinding System (Cement Mill-2) which is located after HPGR and grinds the crushed particles from HPGR. However, after retrofitting of HPGR unit to the front of Cement Mill-2, the grinding media charges of compartments were changed because of reduced feed sizes. Nonetheless, the only difference of Cement Mill-1 and Cement Mill-2 is the sizes of grinding media, and product of Cement Mill-1 also represents the condition of Cement Mill-2 without HPGR support. Thus, that similarity was given a chance to compare the singular ball mill grinding operation and the process of ball mill with roller press while same clinker and gypsum materials was fed to both systems. It was observed that particularly early strengths of COMFLEX® Grinding System and also later strengths were high at same values of sieve residues.

### 4.2 Particle Size Distributions

Particle size distributions of all samples were obtained from PSA with laser diffraction method. Logarithmic distribution curves and histograms of the sampled materials are given in Appendix A.

Narrow and steep histogram curve expresses better classification with properly ground particles. As seen from histograms of products, the HOROMILL® system gave the most efficient results. Individual Roller Press production period was also better than the COMFLEX® Grinding System. When considering COMFLEX® system, the ball mill line was inspected separately and less efficient grinding process was observed. Classifying systems are the main factor of those efficiencies. Moreover, material load of system and air flow speeds with
differential pressures also affect the separating ability of system, and also the grinding performances.

Samples of the returned materials (coarse product after classification) were also inspected with PSA analyses and the results are presented in Appendix B. This is because characteristics of the returned particles are also important to evaluate the performances of classification systems. From this point of view, wide and scattered distributions for returned samples are more reliable if there is a sharp classification. Then, returned particles of HOROMILL® system again supported that situation obviously when graphs are examined.

Furthermore, product samples were also studied with Rosin-Rammler distribution function and graphs were plotted for each product of three grinding systems in Appendix C. It is seen that Rosin-Rammler graphs of HOROMILL® and Roller Press products are steeper than COMFLEX® Grinding System product because of the ball mill products in it. Therefore, steeper lines mean narrower size distribution and effective classification.

4.3 Quality Characteristics of Products

4.3.1 Chemical and Physical Characteristics

Besides the microstructural properties of the cements, chemical compositions were also determined to verify the usage of the same materials at the production periods for each system. XRF analyses were applied for three products, and compositions are given in Table 4.3. Bogue formulation was also used to calculate the clinker phases and modulus, also free calcium oxide, insoluble residue and loss on ignition parameters were measured with other methods, and shown in Table 4.3.

Table 4.3: XRF Analysis Results of Sampled Cements from Grinding Systems

<table>
<thead>
<tr>
<th></th>
<th>SiO₂ (%)</th>
<th>Al₂O₃ (%)</th>
<th>Fe₂O₃ (%)</th>
<th>CaO (%)</th>
<th>MgO (%)</th>
<th>SO₃ (%)</th>
<th>Na₂O (%)</th>
<th>K₂O (%)</th>
<th>Alkali Eq.</th>
<th>CI (%)</th>
<th>Free CaO (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Product of COMFLEX® Grinding System</td>
<td>19.52</td>
<td>4.99</td>
<td>3.63</td>
<td>64.02</td>
<td>1.87</td>
<td>3.22</td>
<td>0.15</td>
<td>0.78</td>
<td>0.66</td>
<td>0.0145</td>
<td>1.98</td>
</tr>
<tr>
<td>Product of HOROMILL®</td>
<td>19.47</td>
<td>4.95</td>
<td>3.68</td>
<td>63.84</td>
<td>1.76</td>
<td>3.14</td>
<td>0.16</td>
<td>0.74</td>
<td>0.65</td>
<td>0.0138</td>
<td>1.95</td>
</tr>
<tr>
<td>Product of Roller Press</td>
<td>19.35</td>
<td>4.96</td>
<td>3.77</td>
<td>63.72</td>
<td>1.85</td>
<td>3.18</td>
<td>0.13</td>
<td>0.75</td>
<td>0.62</td>
<td>0.0132</td>
<td>1.84</td>
</tr>
</tbody>
</table>
Table 4.4: Clinker Quality Parameters, Phases and Other Chemical Analyses

<table>
<thead>
<tr>
<th></th>
<th>Lime Saturation Factor (%)</th>
<th>Modulus of Silica (%)</th>
<th>Mod. of Aluminum (Al/Fe) (%)</th>
<th>$C_3S$ (%)</th>
<th>$C_2S$ (%)</th>
<th>$C_3A$ (%)</th>
<th>$C_4AF$ (%)</th>
<th>Liquid Phase (%)</th>
<th>L.O.I. (%)</th>
<th>Insol. Residue (%)</th>
</tr>
</thead>
<tbody>
<tr>
<td>Product of COMFLEX® Grinding System</td>
<td>100.74</td>
<td>2.29</td>
<td>1.37</td>
<td>54.53</td>
<td>15.51</td>
<td>7.08</td>
<td>11.05</td>
<td>25.94</td>
<td>1.84</td>
<td>0.23</td>
</tr>
<tr>
<td>Product of HOROMILL®</td>
<td>100.75</td>
<td>2.28</td>
<td>1.35</td>
<td>54.68</td>
<td>15.22</td>
<td>6.89</td>
<td>11.20</td>
<td>25.79</td>
<td>1.72</td>
<td>0.22</td>
</tr>
<tr>
<td>Product of Roller Press</td>
<td>100.85</td>
<td>2.25</td>
<td>1.32</td>
<td>54.56</td>
<td>15.05</td>
<td>6.77</td>
<td>11.47</td>
<td>26.09</td>
<td>1.89</td>
<td>0.25</td>
</tr>
</tbody>
</table>

In Table 4.3 and Table 4.4, the sources of produced cements’ performances are clearly illustrated. It is shown that chemical compositions were approximately similar. That investigation is also an acknowledgement of using the same clinker and gypsum in the production period of COMFLEX® Grinding System, roller press and HOROMILL®.

Grinding methods directly affect the physical properties of cements. The same raw materials were ground with fixed Blaine fineness; however, physical characteristics of cement mortars were completely different.

Table 4.5: Physical and Mortar Performances of Sampled Products

<table>
<thead>
<tr>
<th></th>
<th>45µm Residue (%)</th>
<th>Blaine (cm²/g)</th>
<th>Water Demand (%)</th>
<th>Soundness (mm)</th>
<th>Setting Times</th>
<th>Density (g/cm³)</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td></td>
<td></td>
<td></td>
<td>Initial (min)</td>
<td>Final (min)</td>
</tr>
<tr>
<td>COMFLEX® GRINDING</td>
<td>6.1</td>
<td>3683</td>
<td>28.9</td>
<td>2</td>
<td>145</td>
<td>214</td>
</tr>
<tr>
<td>HOROMILL®</td>
<td>3.8</td>
<td>3577</td>
<td>28.6</td>
<td>2</td>
<td>182</td>
<td>293</td>
</tr>
<tr>
<td>ROLLER PRESS</td>
<td>1.8</td>
<td>3656</td>
<td>29.5</td>
<td>1</td>
<td>160</td>
<td>229</td>
</tr>
</tbody>
</table>

From Table 4.5, it can be easily observed that there were dissimilar results of sieve analyses at similar Blaine surface areas. This situation was explained previously by various forces and grinding effects of systems and different particle forms of products. COMFLEX® Grinding System product is a mixture of ball mill and roller press system, and that mixed product has specific characterizations. For instance, sieve residue value for 45 µm is much higher when the Blaine specific surface area is taken constant.

Shapes and forms of particles, surface area variations and internal cracks cause different water requirements. It can be seen that water demand of the Roller Press product was greater than other grinding systems, and that was caused by the microcracked irregular surfaces and higher ratio of finer particles due to
usage of high compressive forces on particles. Product of COMFLEX® Grinding System had the least water demand because it includes uniformly shaped ball mill products.

Soundness values were in the minimum levels for each product as expected, because compositions were similar and there were low free calcium oxide contents.

If setting times are compared, there was an obvious variation between three products. COMFLEX® Grinding System had the earliest initial and final setting periods, also total active setting period (final period – initial period) was the shortest one with roller press product. The main factor of setting is the dehydration conditions of gypsum.

When gypsum (generally dehydrate) is heated, hemi-hydrate and soluble anhydrate is occurred:

\[
\text{CaSO}_4\cdot2\text{H}_2\text{O} \rightarrow \text{CaSO}_4\cdot\frac{1}{2}\text{H}_2\text{O} + \frac{3}{2}\text{H}_2\text{O} \quad \text{(Hemihydrate)}
\]

\[
\text{CaSO}_4\cdot\frac{1}{2}\text{H}_2\text{O} \rightarrow \text{CaSO}_4 + \frac{1}{2}\text{H}_2\text{O} \quad \text{(Soluble Anhydrate)}
\]

The solubility characteristic of the gypsum is related with the form in which it is present in the cement. Gypsum (dihydrate) dissolves slower than hemihydrate and soluble anhydrite. Higher temperatures and longer residence times cause more dehydration process of gypsum. Dehydration rate of used gypsum was also inspected, and illustrated in Figure 4.3.

![Dehydration behavior of the gypsum that was used in the study](image-url)

Figure 4.3: Dehydration behavior of the gypsum that was used in the study
Higher hemihydrates with lower dihydrates improve strength developments, and also increase the effectiveness of gypsum on setting times [19]. It was expected that higher grinding temperatures cause higher dehydration of gypsum, and consequently, proper setting times could be achieved. However, because of different grinding mechanisms, setting periods were not only affected by dehydrated gypsum components but also subsequently affected from changes on reactivity conditions of clinker phases, especially C₃A and C₃S.

Produced cement at COMFLEX® grinding system has the highest temperatures because of ball mill stage in it. Longer residence times with intensive frictional forces cause higher heat generations. However, material contacts and residence times at grinding zones are limited at HOROMILL® and roller press systems. Accordingly, grinding process occurs at lower temperatures and higher air circulations.

As a result, the product of COMFLEX system with an outlet temperature of 104°C set firstly. Then, roller press product, outlet temperature of 92°C, was the second; and the latest setting was occurred with HOROMILL product which had 63°C outlet temperature. It was observed that setting time is shifted with higher grinding temperatures.

On the other hand, there was also a difference between final setting times of the roller press and HOROMILL® products, and relatively total setting periods. Microcracks on the particles of roller press grinding operation bring forward the reactive behavior of cement mortar as defined previously. Accordingly, it is also suggested that microstructural form of the roller press products causes faster decomposition of gypsum’s ettringite formation (which is needle-like formation around preliminary reactive C₃A phase, and retards the hydration process) and accelerates hydration reactions and settings [39].

Density values of products are given in Table 4.5. There are quite differences because of particle shapes and relative compactness of bulk materials. Well-packed ball mill particles increased the specific weights; however, irregularly formed roller press particles covered larger volume.
4.3.2 Compressive Strengths

Alite and Belite crystals are the main strength sources for cement hydration. Especially 5 µm to 30 µm is the most effective size range because hydration reactions are easier [29]. As a result, finer grinding operation improves the performance of cement, and also brings forward the strength developments.

As seen in Table 4.6, early strength of the roller press product is noticeably higher than the other ones. That also caused a 28-days strength advantage for roller press. However, strength development rates (as illustrated in Figure 4.3) show that roller press product has lower 2 to 28 days transition and also 7 to 28 days transition when slopes of those periods compared with other two systems. As a result, effective surface area and microcracked forms also confirmed with strength values, which are reinforced by higher reactivity of cement.

Table 4.6: Compressive Strengths of Sampled Cements

<table>
<thead>
<tr>
<th></th>
<th>1-DAY</th>
<th>Std.Dev.</th>
<th>Averages</th>
</tr>
</thead>
<tbody>
<tr>
<td></td>
<td></td>
<td>1-DAY</td>
<td></td>
</tr>
<tr>
<td>ROLLER PRESS</td>
<td>22.40</td>
<td>23.00</td>
<td>22.60</td>
</tr>
<tr>
<td></td>
<td></td>
<td>22.10</td>
<td>22.53</td>
</tr>
<tr>
<td>COMFLEX® GRINDING</td>
<td>15.80</td>
<td>16.50</td>
<td>16.10</td>
</tr>
<tr>
<td></td>
<td></td>
<td>16.60</td>
<td>16.25</td>
</tr>
<tr>
<td>HOROMILL®</td>
<td>17.00</td>
<td>17.40</td>
<td>16.90</td>
</tr>
<tr>
<td></td>
<td></td>
<td>17.60</td>
<td>17.23</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Std.Dev.</td>
<td>Averages</td>
</tr>
<tr>
<td></td>
<td>2-DAYS</td>
<td>2-DAYS</td>
<td></td>
</tr>
<tr>
<td>ROLLER PRESS</td>
<td>34.70</td>
<td>36.50</td>
<td>35.20</td>
</tr>
<tr>
<td></td>
<td></td>
<td>36.10</td>
<td>35.63</td>
</tr>
<tr>
<td>COMFLEX® GRINDING</td>
<td>27.60</td>
<td>28.40</td>
<td>27.20</td>
</tr>
<tr>
<td></td>
<td></td>
<td>28.20</td>
<td>27.85</td>
</tr>
<tr>
<td>HOROMILL®</td>
<td>28.30</td>
<td>29.00</td>
<td>27.80</td>
</tr>
<tr>
<td></td>
<td></td>
<td>27.70</td>
<td>28.20</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Std.Dev.</td>
<td>Averages</td>
</tr>
<tr>
<td></td>
<td>7-DAYS</td>
<td>7-DAYS</td>
<td></td>
</tr>
<tr>
<td>ROLLER PRESS</td>
<td>48.10</td>
<td>49.40</td>
<td>48.50</td>
</tr>
<tr>
<td></td>
<td></td>
<td>49.90</td>
<td>48.98</td>
</tr>
<tr>
<td>COMFLEX® GRINDING</td>
<td>42.40</td>
<td>41.90</td>
<td>42.50</td>
</tr>
<tr>
<td></td>
<td></td>
<td>41.70</td>
<td>42.13</td>
</tr>
<tr>
<td>HOROMILL®</td>
<td>43.10</td>
<td>43.10</td>
<td>44.20</td>
</tr>
<tr>
<td></td>
<td></td>
<td>42.10</td>
<td>43.13</td>
</tr>
<tr>
<td></td>
<td></td>
<td>Std.Dev.</td>
<td>Averages</td>
</tr>
<tr>
<td></td>
<td>28-DAYS</td>
<td>28-DAYS</td>
<td></td>
</tr>
<tr>
<td>ROLLER PRESS</td>
<td>58.40</td>
<td>57.20</td>
<td>57.50</td>
</tr>
<tr>
<td></td>
<td></td>
<td>58.30</td>
<td>57.85</td>
</tr>
<tr>
<td>COMFLEX® GRINDING</td>
<td>56.50</td>
<td>54.60</td>
<td>55.00</td>
</tr>
<tr>
<td></td>
<td></td>
<td>54.30</td>
<td>55.10</td>
</tr>
<tr>
<td>HOROMILL®</td>
<td>53.40</td>
<td>53.90</td>
<td>53.20</td>
</tr>
<tr>
<td></td>
<td></td>
<td>54.00</td>
<td>53.63</td>
</tr>
</tbody>
</table>
On the other hand, HOROMILL® and COMFLEX® grinding products give ordinary strength performances. Although grinding principles are different, strength values are parallel and transition rates are more regular when compared with Roller Press products.

4.4 Grinding Efficiencies of Systems

4.4.1 Operational Experiences

COMFLEX® Grinding System leads the capacity ranking with 200 tph feeding capacity at a fixed product fineness value of 3600 cm²/g. Feeding rate of the roller press was 95 tph, and that of the HOROMILL® was 48 tph for the production of the same Blaine fineness.

Besides capacities, grinding effectiveness was observed, and COMFLEX® Grinding System was again the better one. At COMFLEX® Grinding System, weakening of pressurized particles and production capability of roller press was supported by uniform grinding performance of ball mill. Although microstructural characteristics affect the water requirements and workability on mortars, that problems could be solved with proper chemical additives and regulations on recipes of concrete mixes.

While producing cement with HOROMILL® system, low grindability (with hardness of clinker) directly affected the performance of mill, and recirculation
ratio was increased. Moreover there were some mechanical limitations of system due to maintenance requirements. Nonetheless, product characteristics and separation sharpness were the operational advantages of the system. Momentary material load was lower than the other systems, and that created more flexible operation with quick responses to operational parameter changes.

Consequences of rough particle forms are more distinct in roller press operations, because there is not a ball mill adjustment. Nonetheless grinding performance and efficiency of system showed that usage of dependent HPGR systems would be a widespread alternative of classical grinding methods. Moreover, singular and parallel operatability is a distinctive property for creating flexibility in maintenance works.

4.4.2 Classifying Efficiency

It is observed that percentages of particles finer than 10 µm were increased at the trial operations of all three grinding systems. This situation is known as fish-hook effect which occurred because of agglomeration of finer particles at the classifying stage. That agglomerated particles got separated at the laser diffraction analysis and caused disorder on the curve. However that fish-hook effect is obviously extensive for HOROMILL® and Roller Press products. On the other hand, in COMFLEX® Grinding System, the agglomeration effect was less than the other systems. This difference was caused by the addition of ball mill product to the roller press product in the COMFLEX® system. Agglomerated particles were separated inside the ball mill because of its grinding mechanism and dissaglomeration effect with mixing materials. This attitude of ball mill operation is also seen from the Tromp curve of ball mill product which was sampled from the COMFLEX® system before mixing the products of roller press and ball mill.

The agglomeration of particles is the result of pressure-based grinding methods which form a cake of particles in the grinding zone. HOROMILL® and Roller Press systems are both working on that principle and the result was experienced in the trials and shown by the curve characteristics.

When the by-pass rates were examined, 35% of the feed material by-passed the separator without classification in the roller press operation as seen from the curve of that system. That by-pass rate was around 20% for other systems, and it was especially related with high feed rate.
Separator efficiency curves are plotted according to capability of selecting finer and coarser particles. Then, the same agglomeration problem at the sampling and analyzing period occurred because of the grinding principles of high compressive forces on caked materials. From the separation efficiency curves, HOROMILL® is seen as less efficient and Roller Press is coming after that. On the other hand, ball mill is seen to have the most efficient separator. However, this evaluation is not correct because the plotted efficiency curves show only the effect of agglomeration on separation. The correct evaluation for the effectiveness of separators is to examine slopes of the Tromp curves. Steeper Tromp curves around cut size $d_{50}$ (between $d_{25}$ and $d_{75}$) show narrow dispersion and sharper separation. From this point of view, HOROMILL® has the highest slope, and then Roller Press is coming. COMFLEX® Grinding System has the lowest slope value because of including ball mill products which were separated less efficiently as seen from the respective Tromp curve. The ball mill product was produced from weakened and pre-classified particles of roller press system; nonetheless, ball mill classification efficiency was the worst one due to older 1st generation separator unit.

4.4.3 Specific Energy Consumptions

4.4.3.1 COMFLEX® Grinding System

Before the installation of the roller press system, the ball mill had a capacity of 65-70 tph while producing CEM I 42.5 R. However, the roller press enhanced the capacity nearly by 300%, and CEM I 42.5 R grinding capacity reached to 205-210 tph. This capacity improvement had been related not only with easy grindability of the pressed and weakened cement particles, but also with the size reduction ability of the roller press. Because the roller press could reduce the particle sizes to the product size ranges, which means that roller press could produce cement individually. By this way, the system capacity increased easily because the coarser particles of the roller press system was returned to the roller press and ball mill feed size became finer.

At the trial operation, composition of CEM I 42.5 R was different from the normal production. Minor component (i.e. limestone) was not added to cement composition, and that caused a harder grindability, which, in turn, caused capacity loss. Feeding capacity was 200 tph (199.7 tph in dry basis) while the sampling was conducted.
Electrical energy consumption measurements were recorded during the period of sampling for the calculation of average hourly consumption after reaching the targeted operational parameters and setting the system to steady state. By the dry basis production of that hour, the specific energy consumption was realized as 30.62 kWh/ton of cement. That specific energy includes not only the main equipment (main drive systems of mill and rollers, system fan motor and separator of roller press unit), but also all the auxiliary equipment like transporting systems (belt conveyors, elevators, air slides); weighfeeders; filter fans and other motors, pneumatic and hydraulic units. However, specific electrical energy consumptions of grinding systems are generally expressed considering only the main equipment such as the drives, system fans and separators. According to that view, specific energy consumption of grinding system was realized as 27.32 kWh/ton cement during the test period. (Table 4.7)

Table 4.7: Equipment-Basis and Total Energy Consumptions for COMFLEX® Grinding System with Operational Parameters

<table>
<thead>
<tr>
<th>COMFLEX® GRINDING SYSTEM</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>MILL-MAIN DRIVE</td>
<td>2240 kWh</td>
</tr>
<tr>
<td>MILL MCC TOTAL</td>
<td>210 kWh</td>
</tr>
<tr>
<td>ROLLER PRESS MAIN DRIVE-1</td>
<td>1370 kWh</td>
</tr>
<tr>
<td>ROLLER PRESS MAIN DRIVE-2</td>
<td>1280 kWh</td>
</tr>
<tr>
<td>ROLLER PRESS SYSTEM FAN</td>
<td>510 kWh</td>
</tr>
<tr>
<td>ROLLER PRESS SEPARATOR</td>
<td>55 kWh</td>
</tr>
<tr>
<td>ROLLER PRESS MCC TOTAL</td>
<td>450 kWh</td>
</tr>
</tbody>
</table>

<p>| | |</p>
<table>
<thead>
<tr>
<th></th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>CAPACITY (DRY)</td>
<td>199.7 tph</td>
</tr>
<tr>
<td>CAPACITY (WET)</td>
<td>200.0 tph</td>
</tr>
<tr>
<td>BLAINE</td>
<td>3683 cm²/g</td>
</tr>
<tr>
<td>Sp. Energy Consumption (Main Equipments)</td>
<td>27.32 kWh/ton</td>
</tr>
<tr>
<td>Sp. Energy Consumption (Total Plant Equipments Basis)</td>
<td>30.62 kWh/ton</td>
</tr>
</tbody>
</table>
4.4.3.2 HOROMILL® Grinding System

HOROMILL® has a design capacity of 85 tph feed rate, and that capacity easily is influenced by the product grindability and moisture factors. At Denizli Cement plant, there are two similar HOROMILL® units. This study was undertaken with the HOROMILL®-2.

Grinding principle of the system is based on cake formation as defined previously. Thus, the pressed material with uniform bedding increases the mechanical efficiency, and that condition is obtained properly while working with additive components like pozzolana because of the ease of grindability and sticky form. Although CEM I 42.5 type of cement has been produced with HOROMILL®, generally blended cements have been produced with HOROMILL® systems at Denizli. However, to compare the grinding systems properly, the same type of cement production with only clinker and gypsum components was selected.

During the test operation, average grinding capacity of the system was 48 tph (47.9 tph on dry basis). After getting first decisive sample, electricity counter values were also recorded. Then, the counter values were recorded again at the end of an hour period, and, thereby, each unit’s electrical energy consumption was obtained. On all-equipment basis 37.25 kWh/ton cement energy was consumed, also for main-equipment basis 33.66 kWh/ton.cement was used to grind cement. (Table 4.8)

Table 4.8: Equipment Basis and Total Energy Consumptions for HOROMILL® System with Operational Parameters

<table>
<thead>
<tr>
<th>HOROMILL® SYSTEM</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>MILL-MAIN DRIVE</td>
<td>1155 kWh</td>
</tr>
<tr>
<td>MCC TOTAL</td>
<td>172 kWh</td>
</tr>
<tr>
<td>SYSTEM FAN</td>
<td>458 kWh</td>
</tr>
<tr>
<td>CAPACITY (DRY)</td>
<td>47.9 tph</td>
</tr>
<tr>
<td>CAPACITY (WET)</td>
<td>48.0 tph</td>
</tr>
<tr>
<td>BLAINE</td>
<td>3577 cm²/g</td>
</tr>
<tr>
<td>Sp. Energy Consumption (Main Equipments)</td>
<td>33.66 kWh/ton</td>
</tr>
<tr>
<td>Sp. Energy Consumption (Total Plant Equipments Basis)</td>
<td>37.25 kWh/ton</td>
</tr>
</tbody>
</table>
4.4.3.3 Roller Press System

Another applicability of the roller press system is its capability of singular operation as explained previously. Rollers crush and reduce the particle sizes, and after a two-stage classification process, appropriately sized products are obtained from the system without ball milling. High pressure forces and efficient classification with proper disagglomeration of pasted particles creates an energy efficient grinding system.

During the test operation of individual roller press unit, the optimum capacity was 95 tph (94.9 tph in dry basis) when the targeted fineness rate was achieved. Energy consumption measurements were executed in a time period of an hour with uninterrupted operation and fixed capacity. The results were calculated as 34.08 kWh/ton.cement for all-equipment basis, and 29.92 kWh/ton cement for main-equipment basis (Table 4.9).

Table 4.9: Equipment Basis and Total Energy Consumptions for Roller Press System with Operational Parameters

<table>
<thead>
<tr>
<th>ROLLER PRESS SYSTEM</th>
<th></th>
</tr>
</thead>
<tbody>
<tr>
<td>MAIN DRIVE-1</td>
<td>1220 kWh</td>
</tr>
<tr>
<td>MAIN DRIVE-2</td>
<td>1150 kWh</td>
</tr>
<tr>
<td>SYSTEM FAN</td>
<td>40 kWh</td>
</tr>
<tr>
<td>SEPARATOR</td>
<td>428 kWh</td>
</tr>
<tr>
<td>MCC TOTAL</td>
<td>395 kWh</td>
</tr>
</tbody>
</table>

| CAPACITY (DRY)       | 95.0 tph  |
| CAPACITY (WET)       | 94.9 tph  |
| BLAINE               | 3656 cm²/g|
| Sp. Energy Consumption (Main Equipments) | 29.92 kWh/ton |
| Sp. Energy Consumption (Total Plant Equipments Basis) | 34.08 kWh/ton |

When the electrical energies are discussed, it is seen that COMFLEX® Grinding System was the most energy-efficient grinding unit. COMFLEX® grinding operation consumed 10.2% less electrical energy than the roller press operation, and 17.8% less than the HOROMILL® operation on the all-equipment basis. When only the main equipment is considered, the corresponding percentages in
the reduction of the consumed electrical energy are 8.7% for the roller press, and 18.8% for the HOROMILL®. The roller press system had a better performance in energy consumption rates than the HOROMILL® system. Nonetheless, better results could be obtained by improving mechanical effectiveness of the system and optimizing operational parameters, and relatively less specific energy consumptions could be provided.

The above results were an account of the operations at fixed Blaine fineness for getting comparable productions. However, in the practice of cement manufacturing, grinding fineness is optimized according to strength performances and workability properties like water demand, soundness etc. Thus, for instance, cement could be produced with higher capacities and lower specific energy consumptions in grinding systems if fixed compressive strength is targeted, and different evaluation results could be realized.
CHAPTER 5

CONCLUSIONS

According to the industrial test productions with three different grinding systems at Denizli Cement Plant (located at Denizli, Turkey) and analyses of the products obtained, the following conclusions could be deduced:

1. Besides the mainly aimed specific energy savings and higher grinding capacities, the grinding technique directly affects the structure of the ground particles, size distributions and specific surface area characteristics of particles in produced cement. Consequently, hydration reactions of cement are obviously affected by the mode of grinding, and favorable specific energy consumptions could be achieved with proper optimizations of the operational parameters (such as fineness) according to strength values.

2. Blaine fineness is not clearly representative for applying comparative analyses of different grinding techniques because of deficiencies of the method with irregular particle structures.

3. The COMFLEX® grinding system is an efficient combination of breaking ability with high compression (roller press) and well-distributed fines with uniform grinding ability (ball mill). This combination provides less specific energy and high production rates. Individual roller press grinding system is an alternative for cement grinding with moderate energy consumptions, but particle characteristics cause disadvantages on service properties like workability, water demand etc. However, high compressive strengths with effective surface area forming ability is the advantage of the roller press; and if service properties are improved properly, roller press could be one of the most effective finish grinding system. On the other hand, HOROMILL® produced a cement product of average quality at higher specific energy consumptions when compared with the other systems. Nonetheless, its classification efficiency and size distribution characteristics are leading properties.
4. At the same Blaine specific surface areas, the roller press product gives the highest compressive strengths, product of the COMFLEX® grinding system is following with moderate strength results, and product of HOROMILL® is third with little difference.

5. HOROMILL® system the best result at classification stages with sharp separation and narrow size dispersion. Roller press system is the next one in classification evaluation, and COMFLEX® system is the third because of less efficient separation at the ball mill stage.
CHAPTER 6

RECOMMENDATIONS

Following recommendations are given for further studies about the investigated industrial grinding systems and their various consequences:

- More effective surface area formations of ground particles in industrial cement grinding operations could be investigated, and savings from specific energy consumptions by optimizing the grinding parameters of systems could be evaluated.

- Research on the relation between shape of cement particles and their effects on service properties of cement and concrete stages could be undertaken.

- Classification efficiencies of air classifiers and the effects of size fractions on the service properties of cement could be investigated in detail. Improving classification efficiency, sharpness and distribution wideness at high efficiency separators need to be studied.

- The study could be improved with research on blended cements, with high proportions of additives, productions with different grinding systems, as in this thesis, and comparing them with the existing results.

- This study could be further substantiated by investigations on dehydration rates of gypsum and its likely effects on the cement products of the grinding systems tested in this thesis work.
REFERENCES


[35] Swenson, E.G., Evans, H.C., Determination of the specific surface area of cements and pozzolans by the Blaine air-permeability method, Division of Building Research, Ottawa, Canada, 1953.


[38] TS EN 197-1, Cement - Part 1: Compositions and conformity criteria for common cements, Turkish Standards Institution, March, 2002.

APPENDIX A

PARTICLE SIZE ANALYSSES OF PRODUCTS

Figure A.1: Particle size analysis of Ball Mill Product in COMFLEX® Grinding System
Figure A.2: Particle size analysis of Roller Press Product in COMFLEX® Grinding System
Figure A.3: Particle size analysis of COMFLEX® Grinding System Product (Mixed Products of Roller Press and Ball Mill)
Figure A.4: Particle size analysis of Roller Press Product (Individual Production)
Figure A.5: Particle size analysis of Horomill® Product
APPENDIX B

PARTICLE SIZE ANALYSES OF TAILINGS

Figure B.1: Particle size analysis of tailings from Ball Mill separator while producing cement in COMFLEX® Grinding System
Figure B.2: Particle size analysis of tailings from Roller Press separator while producing cement in COMFLEX® Grinding System
Figure B.3: Particle size analysis of tailings from Horomill® separator
Figure B.4: Particle size analysis of tailings from Roller Press separator (While on individual production)
APPENDIX C

ROSIN RAMMLER DISTRIBUTIONS OF PRODUCTS

Figure C.1: Rosin-Rammler Distribution Graph of COMFLEX® Grinding System Product

Figure C.2: Rosin-Rammler Distribution Graph of Horomill® System Product

n=1,231

n=1,264
Figure C.3: Rosin-Rammler Distribution Graph of Roller Press System Product

Figure C.4: Comparative Graph of Rosin-Rammler Distributions
APPENDIX D

TROMP CURVES FOR GRINDING SYSTEMS

Figure D.1: Tromp Curve for COMFLEX® Grinding System

Table D.1: Data from Tromp Curve Calculations

<table>
<thead>
<tr>
<th></th>
<th>Slope</th>
<th>Circulating Load</th>
<th>(d_{25}) (µm)</th>
<th>(d_{50}) (µm)</th>
<th>(d_{75}) (µm)</th>
<th>(K = d_{25}/d_{75})</th>
<th>By-pass</th>
</tr>
</thead>
<tbody>
<tr>
<td>Ball Mill</td>
<td>0,627</td>
<td>163%</td>
<td>31,7</td>
<td>74,7</td>
<td>117,7</td>
<td>0,27</td>
<td>19%</td>
</tr>
<tr>
<td>COMFLEX System</td>
<td>1,089</td>
<td>340%</td>
<td>8,9</td>
<td>34,9</td>
<td>60,9</td>
<td>0,15</td>
<td>29%</td>
</tr>
<tr>
<td>Roller Press</td>
<td>1,051</td>
<td>452%</td>
<td>7,4</td>
<td>27,2</td>
<td>47,1</td>
<td>0,16</td>
<td>35%</td>
</tr>
<tr>
<td>HOROMILL</td>
<td>1,123</td>
<td>1556%</td>
<td>15,9</td>
<td>26,9</td>
<td>37,9</td>
<td>0,42</td>
<td>20%</td>
</tr>
</tbody>
</table>
APPENDIX E

SEPARATOR PERFORMANCE AND EFFICIENCY CURVES

Figure E.1: Separator Performance and Efficiency Curves for COMFLEX® Grinding System
Figure E.2: Separator Performance and Efficiency Curves for Ball Mill Operation in COMFLEX® Grinding System
Figure E.3: Separator Performance and Efficiency Curves for Horomill® System
Figure E.4: Separator Performance and Efficiency Curves for Roller Press System (Individual Operation)
APPENDIX F

SCANNING ELECTRON MICROSCOPY ANALYSES OF PRODUCTS

Figure F.1: SEM photographs of COMFLEX® Grinding System Product
Figure F.2: SEM photographs of Horomill® Product
Figure F.3: SEM photographs of Roller Press Product (at individual operation)
Figure F.4: SEM photographs of Ball Mill Product (Denizli Cement Plant-Cement Mill.1)
APPENDIX G

SAMPLING POINTS AT GRINDING SYSTEMS

Figure G.1: Flowsheet of COMFLEX® Grinding System and Sample Points / Roller Press Unit
Figure G.2: Flowsheet of COMFLEX® Grinding System / Rollers

- Feeding Bin
- Fixed Roller
- Moving Roller
- from Feeding + V-Sepator Return
- from Dynamic Separator Return
- to V-Sepator
Figure G.3: Flowsheet of COMFLEX® Grinding System and Sample Points / Ball Mill Unit
Figure G.4: Flowsheet of Horomill® System and Sample Points
Figure G.5: Flowsheet of Roller Press System and Sample
APPENDIX H

SEM ANALYSES OF CLINKER

Figure H.1: Heterogenous Phase Distribution
Alite (brown), Belite (blue), Free Cao (little black zones)

Figure H.2: Alite Crystals with Different Sizes
Figure H.3: Coarse Alite Crystals, Free CaO Sets, Ideal Sized Belite Crystals

Figure H.4: Coarse Belite Crystals with Irregular Shapes
APPENDIX I

FLOWSHEETS OF GRINDING SYSTEMS

Figure I.1: Simple Flowsheet of COMFLEX® Grinding System
Figure I.2: Simple Flowsheet of HOROMILL® Grinding System
Figure 1.3: Simple Flowsheet of Roller Press Grinding System
## APPENDIX J

### DESIGN PARAMETERS OF GRINDING SYSTEMS

Table J.1: COMFLEX® Grinding System Design Data

<table>
<thead>
<tr>
<th><strong>COMFLEX® Grinding System</strong></th>
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</thead>
<tbody>
<tr>
<td><strong>Roller Press</strong></td>
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</tr>
<tr>
<td>Manufacturer</td>
<td>KHD Humboldt Wedag</td>
</tr>
<tr>
<td>Type</td>
<td>RPZ 20/170-180</td>
</tr>
<tr>
<td>Roll Diameter</td>
<td>1,700 mm</td>
</tr>
<tr>
<td>Roll Width</td>
<td>1,800 mm</td>
</tr>
<tr>
<td>Throughput</td>
<td>1,120 tph</td>
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<tr>
<td>Circumferential Speed</td>
<td>1.6 m/s</td>
</tr>
<tr>
<td>Motor Power</td>
<td>1,600 kW</td>
</tr>
<tr>
<td>Number of Motors</td>
<td>2 pcs</td>
</tr>
<tr>
<td>Motor Speed</td>
<td>1,485/min</td>
</tr>
<tr>
<td><strong>V-Separator</strong></td>
<td></td>
</tr>
<tr>
<td>Type</td>
<td>VS 96/26</td>
</tr>
<tr>
<td>Air Flow Rate</td>
<td>328,000 m³/hr</td>
</tr>
<tr>
<td>(at 906 mbar 100°C)</td>
<td></td>
</tr>
<tr>
<td>Height</td>
<td>9,600 mm</td>
</tr>
<tr>
<td>Width</td>
<td>2,600 mm</td>
</tr>
<tr>
<td><strong>Main Separator</strong></td>
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</tr>
<tr>
<td>Type</td>
<td>SEPMASTER® SKS-V 3250</td>
</tr>
<tr>
<td>Motor Power</td>
<td>255 kW</td>
</tr>
<tr>
<td>Adjustable Speed</td>
<td>38 m/s</td>
</tr>
<tr>
<td>Air Flow Rate</td>
<td>328,000 m³/hr</td>
</tr>
<tr>
<td>(at 90°C)</td>
<td></td>
</tr>
<tr>
<td><strong>System Fan</strong></td>
<td></td>
</tr>
<tr>
<td>Air Flow at Inlet</td>
<td>5,450 m³/min</td>
</tr>
<tr>
<td>Dust Load</td>
<td>0.05 g/m³</td>
</tr>
<tr>
<td>Gas Temperature</td>
<td>90°C</td>
</tr>
<tr>
<td>Fan Static Pressure Difference</td>
<td>55 mbar</td>
</tr>
<tr>
<td>Motor Power</td>
<td>700 kW</td>
</tr>
<tr>
<td>Motor Speed</td>
<td>845 rpm</td>
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<tr>
<td><strong>Ball Mill</strong></td>
<td></td>
</tr>
<tr>
<td>Manufacturer</td>
<td>FLSmidth</td>
</tr>
<tr>
<td>Type</td>
<td>Closed Circuit</td>
</tr>
<tr>
<td>Number of Chambers</td>
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<tr>
<td>Diameter</td>
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<tr>
<td>Length</td>
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<tr>
<td>Length Chamber 1</td>
<td>4.0 m</td>
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<tr>
<td>Charge of Chamber 1</td>
<td>17-20-25 mm</td>
</tr>
<tr>
<td>Length Chamber 2</td>
<td>7.5 m</td>
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<td>Charge of Chamber 2</td>
<td>15-17 mm</td>
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<tr>
<td>Mill Speed</td>
<td>16.7 rpm</td>
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<td>Motor Power</td>
<td>2,650 kW</td>
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<td><strong>Mill Separator</strong></td>
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<tr>
<td>Type</td>
<td>REC 450®</td>
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<tr>
<td>Motor Power</td>
<td>110 kW</td>
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Table J.2: HOROMILL® Grinding System Design Data

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<tbody>
<tr>
<td><strong>Mill</strong></td>
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<td>Manufacturer</td>
</tr>
<tr>
<td>Inside Diameter</td>
</tr>
<tr>
<td>Roller Diameter</td>
</tr>
<tr>
<td>Roller/Track Width</td>
</tr>
<tr>
<td>Installed Power</td>
</tr>
<tr>
<td>Mill Shell Speed</td>
</tr>
<tr>
<td>Nominal Pressure (at cylinder)</td>
</tr>
<tr>
<td><strong>Classifier</strong></td>
</tr>
<tr>
<td>Type</td>
</tr>
<tr>
<td>Classifier Body Diameter</td>
</tr>
<tr>
<td>Turbine Diameter</td>
</tr>
<tr>
<td>Nominal Turbine Height</td>
</tr>
<tr>
<td>Number of Blades of the Turbine</td>
</tr>
<tr>
<td>Turbine Rotor Speed</td>
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<tr>
<td>Reducer Reduction Ratio</td>
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<tr>
<td>Installed Motor Power</td>
</tr>
<tr>
<td>Number of Swiveling Counterblades</td>
</tr>
<tr>
<td>Counterblade Drive</td>
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Table J.2: Roller Press Grinding System Design Data

<table>
<thead>
<tr>
<th>ROLLER PRESS</th>
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<tr>
<td><strong>Rolls</strong></td>
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<td>Type</td>
</tr>
<tr>
<td>Roll Diameter</td>
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<tr>
<td>Roll Width</td>
</tr>
<tr>
<td>Throughput</td>
</tr>
<tr>
<td>Circumferential Speed</td>
</tr>
<tr>
<td>Motor Power</td>
</tr>
<tr>
<td>Number of Motors</td>
</tr>
<tr>
<td>Motor Speed</td>
</tr>
<tr>
<td><strong>V-Separator</strong></td>
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<tr>
<td>Type</td>
</tr>
<tr>
<td>Air Flow Rate</td>
</tr>
<tr>
<td>(at 906 mbar 100°C)</td>
</tr>
<tr>
<td>Height</td>
</tr>
<tr>
<td>Width</td>
</tr>
<tr>
<td><strong>Main Separator</strong></td>
</tr>
<tr>
<td>Type</td>
</tr>
<tr>
<td>Motor Power</td>
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<tr>
<td>Adjustable Speed</td>
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<td>Air Flow Rate</td>
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<td>(at 90°C)</td>
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<td>Air Flow at Inlet</td>
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<tr>
<td>Motor Power</td>
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<tr>
<td>Motor Speed</td>
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</table>