# EFFECT OF SOFT FINE PARTICLES ON THE KINETICS AND ENERGETICS OF GRINDING HARD COARSE PARTICLES

A THESIS SUBMITTED TO
THE GRADUATE SCHOOL OF NATURAL AND APPLIED SCIENCES
OF
MIDDLE EAST TECHNICAL UNIVERSITY

BY

SELİM YILMAZ

IN PARTIAL FULFILLMENT OF THE REQUIREMENTS
FOR
THE DEGREE OF MASTER OF SCIENCE
IN
MINING ENGINEERING

DECEMBER 2016

Approval of the thesis:

**EFFECT OF SOFT FINE PARTICLES ON THE KINETICS AND ENERGETICS OF GRINDING HARD COARSE PARTICLES**

submitted by **SELİM YILMAZ** in partial fulfillment of the requirements for the degree of **Master of Science in Mining Engineering Department, Middle East Technical University by,**

Prof. Dr. Gülbin Dural Ünver
Dean, Graduate School of **Natural and Applied Sciences** ___________________

Prof. Dr. Celal Karpuz
Head of Department, **Mining Engineering** ___________________

Prof. Dr. Çetin Hoşten
Supervisor, **Mining Engineering Dept., METU** ___________________

**Examining Committee Members:**

Prof. Dr. Mustafa Ümit Atalay
Mining Engineering Dept., METU ___________________

Prof. Dr. Çetin Hoşten
Mining Engineering Dept., METU ___________________

Prof. Dr. Ali İhsan Arol
Mining Engineering Dept., METU ___________________

Asst. Prof. Dr. İlkay Bengü Çelik
Mining Engineering Dept., HU ___________________

Asst. Prof. Dr. Nurullah Metin Can
Mining Engineering Dept., HU ___________________

**Date:** 15.12.2016

**I hereby declare that all information in this document has been obtained and presented in accordance with academic rules and ethical conduct. I also declare that, as required by these rules and conduct, I have fully cited and referenced all material and results that are not original to this work.**

Name, Last Name: Selim YILMAZ

Signature:

# ABSTRACT

## EFFECT OF SOFT FINE PARTICLES ON THE KINETICS AND ENERGETICS OF GRINDING HARD COARSE PARTICLES



Yılmaz, Selim
M.S., Department of Mining Engineering
Supervisor: Prof. Dr. Çetin Hoşten



December 2016, 75 pages



The main objective of this study is to investigate breakage parameters of a narrow size fraction of coarse particles of a hard mineral when ground in a mixture with fine particles of a soft mineral. For this purpose, quartz and calcite were selected as mixture components varying appreciably in hardness (quartz mohs scale:7 and calcite mohs scale:3) but having quite similar densities. Mixture feeds comprised of -1.18+0.85 mm quartz (hard and coarse) and -106 μm calcite (soft and fine) at various proportions were ground dry or wet in a laboratory batch ball mill for varying times. Besides, in order to delineate the effect caused by the hardness of the fine component in the mixture, a series of similar experiments were also performed with single-mineral mixtures of the coarse and fine size fractions of quartz. Breakage parameters were obtained from the results using the linear batch grinding kinetic model. In addition, using the energy split factor the fraction of specific energy consumed by the coarse quartz when ground in mixture with either calcite fines or quartz fines was compared with that consumed when the coarse quartz fraction was ground alone.

The batch grinding kinetic experiments revealed that breakage distribution is normalizable for all coarse-to-fine ratios. Moreover, it was found that the cumulative breakage distribution function of the coarse quartz fraction remains also unchanged irrespective of whether the fine component in the mixture is fine quartz or fine calcite when ground under identical mill operation conditions. The breakage rate function of the coarse fraction increases as the ratio of the fine (either soft or hard) fraction in the mixture increases. This may be attributed to two reasons: one reason is that a part of the energy applied to particles that is not able to break the finer particles is transmitted to the coarser ones, and the other reason is that larger particles are nipped more easily by the grinding media, and hence exposed to greater number of breakage events. Test results also showed that the calcite fines are less effective than the quartz fines in increasing the breakage rate of the coarse quartz particles. This may be explained by the higher amount of energy absorbed by the soft calcite fines before fracture, and hence leaving less energy available for the breakage of the coarse quartz particles. The energy split factor also supports this finding in the sense that soft fine particles consume more specific energy compared to hard fine particles when ground with hard coarse particles.

Keywords: Mixture Grinding, Soft Material Effect, Ball Mill, Breakage Parameters.

## ÖZ

## YUMUŞAK İNCE TANELERİN SERT İRİ TANELERİN ÖĞÜTME KİNETİĞİ VE ENERJETİĞİ ÜZERİNDEKİ ETKİSİ



Yılmaz, Selim

Yüksek Lisans, Maden Mühendisliği Bölümü

Tez Yöneticisi: Prof. Dr. Çetin Hoşten



Aralık 2016, 75 sayfa



Bu çalışmanın amacı dar tane aralığındaki sert iri malzemelerin yumuşak ince malzeme ile karıştırılıp öğütüldüğündeki kırılma parametrelerini incelemektir. Bu amaçla, aralarında dikkate değer sertlik farkı olmasına rağmen özkütleleri birbirine oldukça yakın olan kuvars (Mohs Sertliği = 7) ve kalsit (Mohs sertliği = 3) karışım malzemeleri olarak seçilmiştir. -1.18+0.85 mm kuvars (sert, iri) ve -106 µm kalsitin (yumuşak, ince) farklı oranlarda karışımından oluşan besleme, kuru veya yaş olarak bilyalı laboratuvar değirmeninde farklı sürelerde öğütülmüştür. Karışımdaki yumuşak, ince malzemenin etkisini karşılaştırmalı olarak belirlemek için, benzer bir dizi deney kuvarsın iri ve ince fraksiyonlarını karıştırarak aynı malzeme için uygulanmıştır. Kırılma parametreleri doğrusal öğütme kinetik modeli sonuçları kullanarak elde edilmiştir. Ayrıca, enerji bölüşümü faktörü kullanılarak iri kuvarsın, yumuşak, ince veya sert, ince malzeme ile karıştırılıp öğütüldüğünde tükettiği özgül enerji fraksiyonu ile tek başına öğütüldüğünde tükettiği karşılaştırılmştır.

Öğütme kinetik testleri göstermiştir ki bütün iri ve ince karışım oranlarında kırılma dağılımı normalize olmaktadır. Ek olarak, iri kuvarsın birikimli kırılma dağılımı ince, sert veya ince, yumuşak karışımların her ikisi için de aynı değirmen çalışma koşulları altında aynı kalmıştır. Karışımdaki ince tanelerin (sert veya yumuşak) oranı arttıkça, iri tanelerin kırılma hızı fonksiyonu da artmıştır. Bu bulgunun nedenleri iki mekanizma ile açıklanabilir: birincisi, malzeme yatağına uygulanan ve ince taneleri kıramayan enerjinin bir bölümünün, iri tanelere aktarılmış olabileceği, diğeri ise iri tanelerin bilyalar arasında daha kolay yakalanabilirlikleri nedeniyle daha fazla kırılma eylemine maruz kalabilecekleridir. Diğer bir bulgu da ince kalsit tanelerinin iri kuvars tanelerinin kırılma hızında neden oldukları artışın, kuvars ince tanelerinin neden olduğu artışa göre daha az olmasıdır. Bu durum, kalsit ince tanelerinin kırılma öncesi kuvars incelerine göre daha fazla enerji absorblayarak iri kuvars tanelerine daha az enerji aktarılması ile açıklanabilir. Enerji bölüşümü faktörü de bu mekanizmayı destekler yöndedir. İnce yumuşak taneler iri tanelerle birlikte öğütülüğünde, sert ince tanelere oranla daha fazla özgül enerji tüketmektedir.

Anahtar Kelimeler: Karışım Öğütme, Yumuşak Malzeme Etkisi, Bilyalı Değirmen, Kırılma Parametreleri.

*To My Lovely Family,*

## ACKNOWLEDGEMENTS

I would like to express deepest appreciation to my supervisor, Prof. Dr. Çetin Hoşten, for his constant invaluable guidance, encouragement and persistent help from the beginning to the end of this research work.

I would also like to thank the members of the examining committee for their kind suggestions and contributions to the content and format of my thesis.

I wish to thank General Manager Sabri Karahan of DAMA Engineering Co. for giving me the opportunity and support to pursue my M.Sc. Degree. I want to thank to Director of Mining and Metallurgy Dr. Tuğcan Tuzcu in DAMA Engineering for his helpful suggestions and encouragement. I would also like to thank my colleagues Okan Vural and Aylin Acar for their friendship and moral support.

I would like to thank my colleague Mahmut Camalan for kindly supporting and guiding me throughout the experimental work.

I would like to thank my friends Gürkan Atalay, Gökhan Yıldırım, Cenk Akın, Cemil Gülen and Serkan Atalay for their friendship and moral support throughout the thesis preparation.

Finally, I would like to give my deepest love to my family who has supported and loved me at every moment of my life.

# TABLE OF CONTENTS

# LIST OF TABLES

TABLES

## LIST OF FIGURES

FIGURES

## LIST OF SYMBOLS

| | |
|---|---|
| D | Internal mill diameter (m) |
| L | Mill length (m) |
| $V_{mill}$ | Empty mill volume ($cm^3$) |
| $N_c$ | Critical mill speed (rpm) |
| $\phi_c$ | Ratio of operation speed to critical speed (%) |
| d | Ball size (m) |
| $d_B$ | Largest ball size used in the mill (mm) |
| $M_{ball}$ | Mass of ball (kg) |
| $\rho_{ball}$ | Ball density ($g/cm^3$) |
| $\varepsilon_{ball}$ | Porosity of the ball bed |
| $\phi_B$ | Volume fraction of ball bed in the empty mill |
| $M_{powder}$ | Mass of powder (kg) |
| $\rho_{powder}$ | Powder density ($g/cm^3$) |
| $\varepsilon_{powder}$ | Porosity of the powder bed |
| $\phi_M$ | Volume fraction of particles in ball porosity |
| $f_c$ | Volume fraction of particle bed in the empty mill |
| E | Energy consumed per unit mass of material (kWh/ton) |
| $X_m$ | Size modulus of Gaudin-Schuhmann |
| $\alpha$ | Distribution modulus of Gaudin-Schuhmann |
| m | mass fractions of grinding component |
| PBM | Population Balance Model |
| $S_i$ | Specific breakage rate of size class “i” ($min^{-1}$) |
| $b_{ij}$ | Individual breakage rate function |
| $B_{ij}$ | Cumulative breakage rate function |
| $w_i(t)$ | Mass fraction of material in the “i” size interval at time “t” in mill |
| W | Mass hold up in the mill |
| $P_i(t)$ | Cumulative mass fraction of material less than upper sieve size of “i” interval at time “t” |

| | |
|---|---|
| P | Net power input to the mill |
| $M_p$ | Mass of particle load into the mill |
| $s_1^E$ | Reduced breakage rate function |
| ES | Energy split factor |
| $s_{1m}$ | Breakage rate function of component 1 in a mixture grinding |
| $s_{1a}$ | Breakage rate function of component 1 when ground alone |
| C:F | Coarse to fine mass ratios in mineral mixture grinding |

# CHAPTER 1

# INTRODUCTION

## 1.1 General

In the material industry, size reduction (comminution) is an essential unit operation and is applied for varying purposes such as to liberate the minerals locked in ore particles or to obtain desired particle size or to increase powder surface area. In the mineral processing industry, the comminution process is generally conducted for the purpose of liberation of a mineral of interest from the associated gangue, after which separation of the valuable mineral grains from the gangue minerals, by utilization of the physical and chemical properties of the component, is achievable.

It is commonly known that size reduction, particularly at the finer end, is an energy intensive process and that most of the energy consumption in mineral processing plants is addressed to this unit operation. An investigation shows that comminution accounted for approximately 29.3% of total mining energy consumption in the U.S. This consumption corresponds to 1.14% of the total energy used by the industrial sector and 0.39% of the overall energy usage in the U.S. (Tromans, 2008). Although comminution is known as the most energy consuming unit operation, utilization of energy in a grinding mill is extremely inefficient (Austin et al., 1984), with a major part of mill energy lost in machinery movement, heat, sound etc. Hence, better understanding and description of the comminution process has a vital role to enhance the proper use of energy.

Comminution is mostly achieved by mechanical crushing and grinding operations. These two steps are usually performed in sequence, with crushers taking large chunks and breaking down the particle size to some degree; the products of which considered as relatively coarse. As the most valuable minerals are finely embedded in the ore, grinding processes that provide fine material are generally required. Different types of grinding machines exist, such as rod mill, ball mill, autogenous mill, semi-autogenous mill, pebble mill, high pressure grinding rolls etc. Among these, the ball mill is most extensively used in industry (Acar, 2013).

Grinding has been studied over the years and some scientific models have been developed to describe the unit operation. The first models developed by researchers were based on the interrelation of specific energy input to the mill and size reduction ratios. Since those approaches do not take into account the breakage kinetics, size distribution and some subprocesses of grinding, they have been found inadequate for simulation and scale up of the mill (Herbst and Fuerstenau, 1973,1980). Therefore, improved mathematical models have been adapted which are derived from population balance models (PBMs). Those models consider two main parameters which are the breakage rate (selection) function and the breakage distribution function, used in order to examine grinding kinetics of the particulate environment. Those parameters are important for predicting the entire size distribution in grinding processes.

Initially, single size pure material was ground and investigated to make inferences about grinding of the naturally occurring size distributions. This is called the batch grinding approach and gives reasonable results, but for industrial milling generally the requirement is to ground heterogeneous materials with different compositions. Thus, it was necessary to determine and understand the effects of different materials on one another in the grinding environment. Therefore, scientists started to study grinding of heterogeneous material which were generally based on mixtures of particles of initially the same size but composed of different materials (Fuerstenau and Sullivan, 1962; Somasundaran and Fuerstenau, 1963; Venkataraman and Fuerstenau, 1984; Kanda et al., 1989) or different sizes of the same material (Austin and Bagga, 1981, Celik, 1988; Fuerstenau and Abouzeid, 1991; Fuerstenau et al., 2010).

## 1.2 Objective and Scope of Thesis

The objective of this study was to determine how the addition of fine particles of a soft mineral to closely-sized coarse particles of a hard mineral affects the breakage rate and breakage distribution functions of the hard and coarse particles. For this purpose, quartz and calcite were chosen as minerals varying appreciably in hardness but of quite similar densities. Feeds consisting of closely-sized (-1.18+0.85 mm) coarse particles of the hard mineral (quartz) with various proportions of the fine particles (-106 μm) of the soft mineral (calcite) were ground dry or wet in a batch laboratory ball mill for varying times and the breakage parameters were obtained from the results using the linear batch grinding kinetic model. In order to delineate the effects caused by the hardness of the fine component in the mixture, a series of similar experiments were also performed with mixtures of the coarse and fine size fractions of quartz. In addition, experiments were conducted with single size fractions of calcite to determine its breakage parameters in the coarse (-1.18+0.85 mm) and fine (-150+106 μm) size ranges. Experimental results were then interpreted to investigate the effect of soft, fine particles on the breakage parameters of hard, coarse particles in mixture grinding. Using the concept of energy split factor (Kapur and Fuerstenau, 1988), the specific energy consumed by the coarse quartz fraction when ground in mixtures with either calcite fines or quartz fines was compared with that consumed when the coarse quartz fraction was ground alone. Hopefully, this study will be helpful to better understand the industrial grinding of heterogeneous mixtures to maximize grinding efficiency and reduce energy input.

# CHAPTER 2

# BACKGROUND

## 2.1 Comminution in Mineral Processing

### 2.1.1 General

In nature, materials generally occur as assemblies of mineral mixtures having different physical and chemical properties. Those compounds necessitate liberation for subsequent concentration processes. The liberation of a specific material is carried out by comminution in which the particle size of the material is progressively reduced until the desired size of material is obtained (Wills and Napier-Munn, 2006). Comminution is accomplished by two primary stages which are crushing and grinding. The larger particles coming from the mine site are firstly fed to crushers. Crushers' output are usually not fine enough to liberate the minerals so that further size reduction is generally required. This can be achieved by grinding operation (or milling) at which very fine particles can be produced. A number of different mill types have been developed for specific grinding requirements. However, the most widely used grinding equipment in the mineral processing industry is the ball mill.

### 2.1.2 Ball Mill

A ball mill is a cylindrical steel vessel a large proportion of whose volume is charged with balls. The internal surface of the steel cylinder is generally covered with renewable liners and equally spaced lifter bars are mounted along the length of the cylinder. Two principal breakage mechanisms exist in the ball mill to break down the material. The first one is impact, which can be supplied by rotation motion of the mill around its horizontal axis, by which the material and the balls within the mill with the help of lifter bars are raised to a certain height before falling down on the ball bed.

The material nipped between two balls, or between balls and walls are subject to impact breakage. The second mechanism is abrasion, which arises in a way that some part of the balls and material mixture during rotation are not able to rise and slide down from the wall, and this contact of material between balls results in abrasion breakage. Figure 2.1 illustrates the operation of the ball mill.

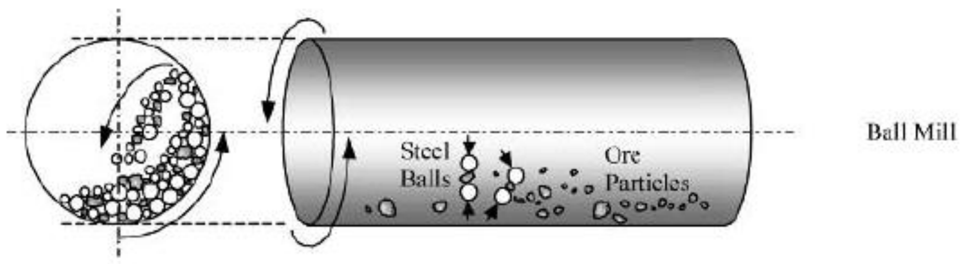


Figure 2.1 Representation of the principle of operation of a ball mill (Gupta and Yan, 2006)

The size reduction in the ball mill results from repetitive breakage actions of impact and abrasion. The material entering the ball mill commonly has a complete size distribution and leaving it again has an entirely different size distribution after being reduced down in size. Ball mills are usually operated in wet conditions, although some dry grinding operations take place.

The analysis of particle breakage in a ball mill is quite a complex task. The fragments produced by a ball mill are influenced by a number of factors related to both the material characteristics and the mill environment. Apart from the material characteristics, the efficiency of the ball mill greatly depends on how it is operated. Thus, some of the operational parameters are defined below:

The critical speed is a rotational speed at which gravitational forces on balls at the top of the mill equals to centrifuge forces as formulated in Eq. (1); above this speed balls start to centrifuge on the mill shell and no milling occurs.

$$N_c(rpm) = \frac{42.2}{\sqrt{D - \mathrm{d}}} \qquad (1)$$

where D and d are the internal mill diameter and the largest ball diameter in meters, respectively. The rotational mill speeds are typically expressed as a fraction of the critical speed, $\phi c$. Most mills are run at 50-90% of their critical speed (Wills and Napier-Munn, 2006).

The other operational parameter is fractional ball filling, $\phi_B$ which is defined as the fraction of the mill volume occupied by ball charges including voids between the ball bed at rest. This is given by Eq. (2):

$$\phi_B = \frac{M_{ball}/\rho_{ball}}{V_{mill}} x \frac{1}{(1 - \varepsilon_{ball})} \qquad (2)$$

where $M_{ball}$ and $\rho_{ball}$ are the overall mass of the balls and the density of a ball, respectively. $V_{mill}$ is the volume of the mill and $\varepsilon_{ball}$ is ball bed porosity fraction. The porosity between the single size ball bed is commonly taken as 40%. In a same way, filling of powder, $f_c$, is defined as the fraction of mill volume occupied by powder including voids between powders. This is given by Eq. (3):

$$\mathrm{f}_c = \frac{M_{powder}/\rho_{powder}}{V_{mill}} x \frac{1}{(1 - \varepsilon_{powder})} \qquad (3)$$

where $M_{powder}$ and $\rho_{powder}$ are the mass of the material used to filled the mill and the density of the material, respectively. $V_{mill}$ is the volume of the mill and $\varepsilon_{powder}$ is powder bed porosity fraction. The material filling is also expressed as the fraction of volume occupied by powder, $\phi_M$, in void space between the balls which is formulated as shown in Eq. (4) below:

$$\phi_M = \frac{f_c}{\varepsilon_{ball} * \phi_{ball}} \tag{4}$$

Keeping $\phi_M$ in the range of 0.6 to 1.1 gives a better grinding result (Austin et al., 1984).

## 2.2 Energy Related Laws of Grinding

It is commonly stated by scientists from the past to today that comminution is an energy inefficient process. The energy applied to particles to fracture them is transferred to cracks and this induces the breaking up of the particle. The study of single size fracture with a given load can be found in the literature (Anderson, 2005). However, in the ball mill, since the particle undergoes a range of impact forces by multi-ball collisions, to analyze fracture processes from a fundamental point of view is almost impossible (Verma and Rajamani, 1995). Thereby, previous studies investigating grinding mills to elucidate size reduction process were mainly on the basis of energy given to the comminution device. There were lots of scientific attempts to explain energy-size reduction relations on scientific basis. As a result of those attempts, a number of fundamental grinding laws have been developed. Walker et al. (1937) (quoted from Austin et al., 1984) claimed that those laws originated from the general law of energy-size reduction given in Eq. (5) below:

$$dE = -\frac{C}{x^n} dx \tag{5}$$

which reflects that the specific energy required (dE) to reduce the particle size is directly proportional to the particles size change (dx) and inversely proportional to the single size of the material (x) to some power n (Charles, 1957). There are three widely accepted grinding laws in the literature. The equations of the grinding laws of the those researchers can be obtained by integration of the general law of energy-size reduction (5) by substitution of the following n values;

n: 2 (Rittinger's law)

n: 1 (Kick's law)

n: 1.5 (Bond's law)

Those researchers each considered energy-size reduction relationships in terms of a different aspect. Rittinger's theory (1867) states that the energy used for particles breakage is proportional to the creation of new surface area per unit volume (Fuerstenau and Abouzeid, 2002). According to Kick's theory (1885), energy consumption is determined by the reduction in volume of the particles size (Fuerstenau and Abouzeid, 2002). In the case of Bond's grinding theory (1952), the energy required for size reduction is proportional to the length of crack formed (Wills and Napier-Munn, 2006).

For those grinding laws, particle (single) size x is expressed as representing the feed or product size distribution so that it is generally taken as the value of mean particle size or 80% passing size of the distribution.

Similar to the other grinding laws, Charles' law is derived from the above equation of the general grinding law and takes the form:

$$E = CX_m^{-\alpha} \tag{6}$$

where E is the energy consumed per unit mass of material (kWh/ton), C is a constant for that particular mill and material system, $X_m$ is the size modulus of Gaudin-Schuhmann and α is the distribution modulus of Gaudin-Schuhmann.

## 2.3 Mathematical Models in Comminution

Energy size reduction models developed at the early stage of investigation of comminution mostly deal with the interrelation of a single (particle) size measure of the product and the specific energy input to the mill. Although those models provide a good correlation for the results of experimental data, they are unable to produce adequate information to apply simulation and optimization processes for comminution (Herbst and Fuerstenau, 1973,1980). Therefore, mathematical models have been adapted to comminution process by which the relation of the size distribution of the particles entering and leaving the comminution devices can be estimated, enabling quantitative simulation of the operation. In the comminution process, feed particles undergo a number of repetitive (or time period) breakage actions and each action generates a complete size range of products. The mathematical models based on a mechanistic approach to the size reduction process, via a set of relations between feed and product size distributions and analysis of the variation of those components during comminution. A method known as Population Balance Model (PBM) is used for mass balance over a range of sizes between feed and product. This model is used efficiently to analyze comminution mathematically; however, it has no direct information on the underlying physics of the process, and thus it is called a phenomenological model.

There are three basic forms of PBM available in the literature for grinding models. One of the forms is the size-continuous time-continuous model. This model requires mass balances on differential size intervals, resulting in an equation for which an analytical solution is almost impossible as yet (Phatak, 1990). Given the unlikeliness of a solution to the size-continuous models, size discrete models were also developed, including:

- Discrete-size discrete-time model (Matrix Model)
- Discrete-size continuous-time model (Kinetic Model)

In these models, the feed and product are divided into a number of discrete size intervals and then mass balances are performed. The matrix model considers comminution as a discrete process, which consists of repeated breakage-selection-classification cycles, thus time is implicitly defined in the model. Since time is implicit in the matrix model it is more likely applicable to devices in which residence times are fixed. The model details are available in the literature (Lynch, 1977). On the other hand, the kinetic model considers comminution as continues process and time is explicit in the model. The kinetic model (discrete-size continuous-time model) has been proven quite useful in describing most comminution systems (Fuerstenau et al., 2011).

### 2.3.1 Kinetic Model

The kinetic approach (size-discrete time-continuous model of PBM) has been widely and successfully used to analyze the comminution in a tumbling mill. This model depends on two fundamental concepts which are:

- Specific Rate of Breakage (Breakage Rate or Disappearance function)
- Breakage Distribution Function (Primary Progeny Distribution)

**Specific Rate of Breakage (Breakage Rate of Disappearance Function)**

Particles entering and leaving the grinding mill have a complete size distribution. In the kinetic approach, the range of particle sizes is split into a set of intervals with a geometric progression, with the top size interval defined as 1 and the following intervals as 2, 3 etc. The particles in the $i^{th}$ size interval are bounded by i and i+1 sizes. The specific rate of breakage, $S_{i,}$, is defined as the fraction of material weight in the $i^{th}$ size interval broken out per unit time. In other words, disappearance of the mass fraction of material in the size “i” interval which is selected to break per unit time “t”. A number of single size batch grinding test results showed that the rate of disappearance of homogenous materials follows the first-order law shown in the following equation (Austin et al., 1984).

Rate of disappearance of size i ; $-\frac{dw_i(t)}{dt} = S_i w_i(t)$ (7)

where $S_i$ is a selection or disappearance function and has a unit of time$^{-1}$, $w_i(t)$ is the mass fraction of material in the i$^{th}$ size interval at time "t". If the breakage rate, $S_i$, is independent of time, then the integration of Eq. (7) becomes:

$$w_i(t) = w_i(0)\exp(-S_i t) \quad (8)$$

or

$$\log w_i(t) = \log w_i(0) - \frac{S_i t}{2.303} \quad (9)$$

Considering that $S_i$ is constant with time, the specific breakage rate can be determined from the slope of $w_i(t)/ w_i(0)$ versus time on a semi-log plot. This plot for a single size fraction commonly gives a straight line which implies that the breakage rate follows the first-order hypothesis. Although this is the case commonly observed in a milling environment for a single size fraction, it does not necessarily mean that finer materials demonstrate first-order breakage in the presence of varying quantities of a coarser material (Austin et al., 1984). On the other hand, in the case of milling of relatively coarser particles, deviation from first-order kinetics is frequently observed due to the fact that the particle sizes are too big for the grinding media to nip properly (Austin et al., 1981), which is called abnormal breakage

The specific rate of breakage depends on the properties of the material being ground as well as mill variables such as the mill size, the filling ratio, the rotational speed, the ball size and the load, etc. The material breaks according to the first-order kinetics rule initially, but after some point can show non-first-order behavior due to a number of the reasons arising from material variation or environmental changes in the mill (Figure 2.2). The possible reasons for non-linearity are given by Austin et al. (1984) as follows:

- The stronger fraction of the material might be accumulated in the mill as grinding proceeds leading to a decrease in breakage rate
- The fine particles in a mixture environment can act as a cushion for breakage of the coarser particles, leading to deceleration of the breakage rate.
- An increase of fine material in the mill environment can affect the milling mechanism which results in less number of particles lifting and thus a decrease in the breakage rate.
- The fine particles in the mill environment might agglomerate causing an increase of the amount of larger particles, which decreases the breakage rate.
- The material weakens at first, and is thus more easily broken as grinding proceeds which leads to an increase of the breakage rate.
- The heterogeneity in mineral composition can cause an increase of the breakage rate such that grinding of the harder component at first accelerates the softer one later in the process.
- Coarser particles have a larger cross section and may behave like a shield for the smaller particles; removal of those particles can increase the breakage rate.

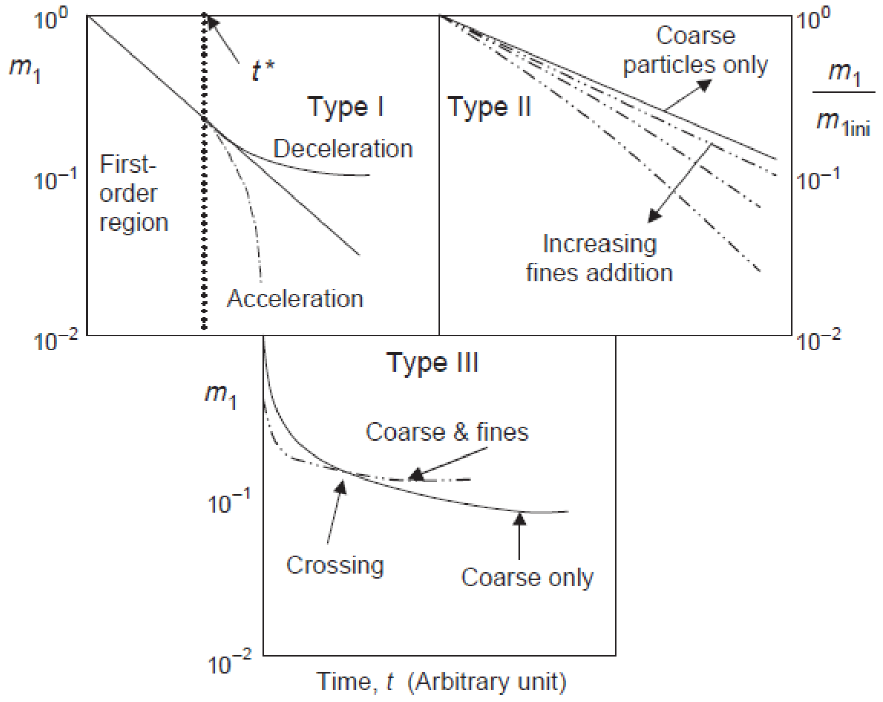


Figure 2.2 Illustration of potential deviations of the breakage rate from linearity during milling (Bilgili and Scarlett, 2005).

**Primary Breakage Distribution Function (Progeny Fragment Distribution)**

The primary breakage distribution function, $b_{ij}$, is a set of primary daughter fragments produced by primary breakage. Breakage in the mill is a continuous process such that the fragments produced by breakage will be subject to re-breakage together with the rest of the mill content. The observation of fragments of distribution before re-selection for further breakage gives the primary breakage distribution function. It should be recognized that primary breakage distribution does not indicate the distribution produced by a single breakage; instead it means that the distribution produced by breakage actions need to be measured before remixing with mill content.

The size fraction of broken particles in different sizes is designated by $b_{ij}$ elements, that shows the fraction of broken particles of the size interval "j" which fall into the interval "i". In here, the breakage action is realized if the material is broken and appears in a finer size class, thereof the size class “i” should be smaller than the size class “j”. By definition, the material broken from size interval 1 goes to the 2nd size interval and is designated by $b_{21}$ while the material from size internal 1 which goes to the 3rd size interval is indicated by $b_{31}$ and which goes to 4th size interval by $b_{41}$ and so on.

By definition, the sum of the mass fractions of any material which appears in the other sizes should be equal to 1, which is shown by Eq. (10).

$$\sum_{i=j+1}^{n} b_{i,j} = 1 \tag{10}$$

Table 2.1 shows the breakage distribution size range split into n discrete sizes with top size interval 1 and the nth interval being the sink containing all finer material.

Table 2.1 Illustration of breakage distribution function in a matrix form

| | j=1 | j=2 | j=3 | j=4 | j=5 | j=6 | j=n |
|---|---|---|---|---|---|---|---|
| i=1 | 0 | 0 | 0 | 0 | 0 | 0 | 0 |
| i=2 | b21 | 0 | 0 | 0 | 0 | 0 | 0 |
| i=3 | b31 | b32 | 0 | 0 | 0 | 0 | 0 |
| i=4 | b41 | b42 | b43 | 0 | 0 | 0 | 0 |
| i=5 | b51 | b52 | b53 | b54 | 0 | 0 | 0 |
| i=6 | b61 | b62 | b63 | b64 | b65 | 0 | 0 |
| i=n | bn1 | bn2 | bn3 | bn4 | bn5 | bn6 | 0 |

The breakage distribution function is often used as a cumulative form which is shown using the symbolism of $B_{ij}$. This represents a cumulative mass fraction of material less than the upper size of interval “i” resulting from breakage of size interval “j”. The $B_{ij}$ function can be formulated as shown in Eq. (11):

$$B_{i,j} = \sum_{\substack{k=j \\ i>j}}^{n} b_{i,j} \tag{11}$$

Where;

$$b_{i,j} = B_{i,j} - B_{(i+1),j} \tag{12}$$

and by definition

$$B_{j+1,j} = 1 \; and \;\; B_{n,j} = b_{n,j} \tag{13}$$

A number of experimental results show that the breakage distribution function $b_{ij}$ remains relatively invariant irrespective of changes in the mill environment conditions for any given material. Moreover, it is commonly observed from experiments that $b_{ij}$ is dimensionally normalizable. In other words, breakage of all particle sizes gives the same progeny distribution function based on relative sizes (Austin et al., 1984).

### 2.3.2 Batch Grinding Equation

In an operating mill, the breakage of particles is continuous and is performed on the whole range of particle size distributions which are exposed to re-breakage. The concept of selection (breakage) function, $S_i$, and breakage distribution function, $b_{ij}$, explained above can be used to model the milling operation mathematically, and thus they allow a complete mass balance of the entire size range, which is divided into n number of size class, to be performed, as shown in Eq. (14).

$$\frac{d[w_i(t)W]}{dt} = -S_i w_i(t)W + \sum_{\substack{j=1 \\ i>1}}^{i-1} b_{ij} S_j w_j(t)W \tag{14}$$

Equation (14) expressed that concept that the net rate of production of the material in the size interval "i" equals the sum of the rates of disappearance of size "i" by breakage and appearance of size "i" from breakage of larger sizes. The rest of the symbols has same meaning as given in Eq. (7) and Eq. (11)

Considering the case where the mill content "W" is constant with time, then Eq. (14) becomes;

$$\frac{dw_i(t)}{dt} = -S_i w_i(t) + \sum_{\substack{j=1 \\ i>1}}^{i-1} b_{ij} S_j w_j(t) \qquad (15)$$

Eq. (15) inherently includes some of the assumptions given by Austin et al. (1984) such that;

- It does not account for minor breakage or chipping, $b_{ii}$=0;
- There is no re-growth of the particles by cold welding;
- The materials contained in a specific interval have identical fracture properties irrespective of whether they arrived through breakage of other sizes or were initially this size;
- The material being ground is homogenous such that it does not show any difference in grinding behavior although it is composed of a mixture. Actual tests performed on mineral mixtures show linear grinding behavior, meaning that the assumption of material homogeneity is not strictly valid.

## 2.4 Determination of Breakage Functions

Most mathematical grinding equations are built on basically two parameters which are breakage rate function and breakage distribution function (Rajamani and Guo, 1992). Thus, determination of those parameters has priority for application of these equations. The accurate estimation of breakage rate function and breakage distribution function is vital to applying the grinding equation for simulation, scale-up and optimization purposes. There are two main techniques to estimate the breakage rate and breakage distribution parameter: the first is direct estimation of the parameters from experimental data, the other is a back-calculation technique from batch and continuous systems. Back calculation methodology is not discussed in this thesis; the interested reader can refer to the book of Austin et al. 1984.

### 2.4.1 Direct Determination of Breakage Functions

The breakage rate function (selection function) and breakage distribution function (primary progeny function) can be obtained directly from batch laboratory tests which are based on grinding of narrowly-sized (one size fraction) materials generally set with a $\sqrt{2}$ sieve size interval. The application of this experiment starts with preparation of a suitable amount of a one size fraction material as feed to the mill. The mill is filled with material and the grinding media and ground for set of times. After each grinding time, the mill is stopped, discharged and the material separated from the grinding media. The obtained product is sampled and subject to sieve analysis. It is then returned to the mill for further grinding, or instead a fresh feed can be used for longer grinding times.

Only analysis of the material retained in the lower screen of the top size interval for each discrete time is adequate to obtain the breakage rate function (selection function), $S_i$. On the other hand, determination of the breakage distribution function, $b_{ij}$, requires complete size analysis data at short grind times.

As previously discussed, experimental work has shown that the rate of disappearance of the homogenous material in the geometric size interval ($\sqrt{2}$) follows a first-order law. If this is the case, a semi log plot of the weight percent remaining in the top size versus grinding time gives a straight line (Figure 2.3); from the slope of this line the breakage rate can be determined.

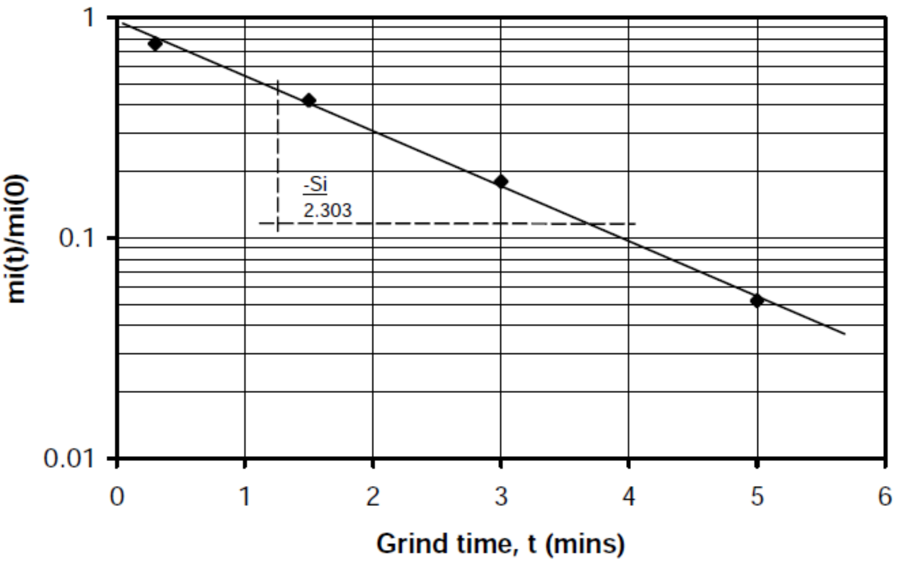


Figure 2.3 First order plot for breakage rate determination (Gupta and Yan, 2006).

The size analysis of the material for determination of the breakage distribution function needs to be performed at short grind times in order to hold re-breakage of the smaller material at a low level. However, it is difficult to get accurate size analysis result with this small degree of breakage. Austin and co-workers proposed a method called the BII method (Austin et. al., 1984) which is based on use of batch grinding data. This method uses size distribution produced by a single √2 size fraction material and allows re-breakage of the progeny distribution by assuming that the product of the breakage rate function and the breakage distribution function, $S_jB_{ij}$, is approximately constant. Thus, the equation for those conditions is given in Eq. (16) below:

$$1 - P_i(t) \cong [1 - P_i(0)]exp(-B_{i1}S_1t), \quad i>j \qquad (16)$$

For the top size interval, first order breakage gives

$$1 - P_2(t) = [1 - P_2(0)]exp(-S_1t), \quad i>j \qquad (17)$$

Then,

$$-S_1 t = \ln[(1 - P_2(t))/(1 - P_2(0))] \tag{18}$$

$$-B_{i,1} S_1 t \cong \ln[(1 - P_i(t))/(1 - P_i(0))] \tag{19}$$

and

$$B_{i1} = \frac{\log\left(\frac{1 - P_i(0)}{1 - P_i(t)}\right)}{\log\left(\frac{1 - P_2(0)}{1 - P_2(t)}\right)} \tag{20}$$

where;

$P_i$ = cumulative mass fraction of material less than size i at a specific time,

$B_{ij}$ = cumulative mass fraction of material that goes to size i upon breakage of size j.

### 2.5 Energy Split Factor

Initial efforts to develop energy size reduction relationships were generally completed based on studies conducted on single size pure minerals, which is rather reasonable when considering the complexity of mixture grinding. However, most of the material ground in industrial mills is composed of mineral mixtures, and this single size of pure material approach underestimates the effects of different materials have on each other in a grinding environment. One of the first attempts to comprehend mixture grinding was performed by Fuerstenau and Sullivan (1962). They ground a quartz-limestone material mixture in a ball mill, and by analysis of the result they reported that the energy required for grinding of a unit mass of material mixture is the sum of the energy expended for grinding each component which is formulated as shown in Eq. (21) below using Charles' grinding law (Section 2.2) (Kapur and Fuerstenau, 1988).

$$E_t = m_1 C_1 X_{m_1}^{-\alpha_1} + m_2 C_2 X_{m_2}^{-\alpha_2} \tag{21}$$

where $E_t$ is the energy consumed per unit mass of material mixture (kWh/ton), and 1 and 2 denote the components of the mineral mixture, m and α are mass fraction and the distribution modulus of each component, respectively. C is a constant for the mill and mineral system for single size grinding, and $X_m$ is the size modulus of the grinding component for the mineral mixture. The authors also stated that the energy expended by quartz and limestone is proportional to their mass fraction in a ball mill. This means there is no preferential grinding, a finding which is questioned with later studies. Somasundaran and Fuerstenau published a study in 1963 and postulated that energy consumption of any material in the mill is proportional to its volume fraction but to a limited extend and that preferential grinding does occur.

Herbst and Fuerstenau (1973) dry ground mono-sized dolomite in a laboratory ball mill under varying operating conditions and observed from the results of these experiments that the ratio of breakage rate and specific power is constant. This constant was called the reduced breakage rate function given by the following formula (Equation 22):

$$s_1 = s_1^E \left(\frac{P}{M_p}\right) \tag{22}$$

where $s_1^E$ is the reduced breakage rate function, P is the net power input to the mill, $M_p$ is the mass of particle load. This concept actually introduces the idea that breakage rate is proportional to the specific power applied. Venkatraman and Fuerstenau (1984) ground binary mixtures of hematite, quartz and calcite and concluded from the experiments that the reduced or normalized breakage function concept is valid for multicomponent grinding although originally developed for pure minerals. Hence, according to them "the first-order breakage kinetics can be normalized in terms of specific energy for the minerals even in mixture grinding".

In 1988, Kapur and Fuerstenau published a study whose purpose was to integrate the energetic and kinetic aspects of multicomponent feed grinding and proposed an appropriate formula for this relationship. The observation that the grinding path of the material did not show any difference whether comminuted alone or as a component of mixtures led them to develop a power-breakage rate correlation (Eq. 22) to integrate kinetics and energetics of the mixture grinding. They introduced the energy split factor concept which is defined as the ratio of energy expended when a unit mass of a mineral is ground as a component in a mixture and alone for the same time interval. This is given by Eq. (23):

$$ES_1 = \frac{E_{1m}}{E_{1a}} \tag{23}$$

where ES is the energy split factor and E is the energy expended per unit mass of material. 1 denotes for component 1 in the mineral mixture, subscripts a and m represent the material ground alone or in a mixture, respectively. Then, to determine the energy split factor from the experimental data, the authors compared the breakage rate function of the material when it was ground alone or as a mixture component. Finally, they expressed the energy split factor as if the breakage rate function does not depend on time, as shown in Eq. (24):

$$ES_1 = \frac{s_{1m}}{s_{1a}} \tag{24}$$

where ES is the energy split factor and s is the breakage rate function. The rest of the symbols have the same meaning as given in Eq. (23). Likewise, for time-dependent breakage rate function energy split function can be calculated with formula

$$ES_1(t) = ln\left[\frac{m_{1m}(0)}{m_{1m}(t)}\right] \Big/ ln\left[\frac{m_{1a}(0)}{m_{1a}(t)}\right] \tag{25}$$

where ES is energy split factor and m is mass fraction at grind time t and initial condition, and the rest of the symbols has same meaning as given in Eq. (23).

## 2.6 Previous Studies for Mixture Grinding

Attempts to observe the kinetics of size-discretized single size pure minerals with breakage parameters in order to make inferences about grinding behavior of naturally sized feeds of industrial mill were found to be quite successful. After those valuable attempts, researchers started to analyze heterogeneous environments that are more representative of industrial grinding in which mineral mixture grinding occurs in terms of particle type and size. Some of the studies related to heterogeneous grinding are summarized below.

Austin and Bagga (1981) dry ground single sizes of several cement clinkers and two different coal materials in a laboratory tumbling ball mill. They reported that the breakage rate functions of tested materials (40x50 or 20x30 or 16x20 mesh) follow first-order kinetics initially, but start to slow down as the fine particles accumulate in the mill. This was also observed for all sizes in the mill. In order to clarify this finding, breakage rates of 16x20 mesh materials in 1:1 mixture with -200 mesh materials were ground and investigated. It was postulated that although the power input to the mill did not differ significantly for tests, the breakage rate of the coarse material slowed down. The reason why the breakage rate decreased for all sizes was explained by the cushioning action of the fine particles.

Venkataraman and Fuerstenau (1984) conducted a set of single size (10x14 mesh) batch dry grinding experiments with quartz, hematite and calcite, individually and as binary mixtures. They observed from the experiments that disappearance kinetics plots for dry grinding single size fractions of the materials follow the first-order law whether ground alone or as binary mixtures. Additionally, they found that soft particles grind faster in 1:1 mixtures of hard and soft components of the same size than when they are ground alone, and indicated the reverse was true for the harder component. Besides, they also stated that the breakage distribution function did not show any difference whether comminuted alone or as a component of mixtures. Thus, they concluded that the breakage rate function is time-independent but environment dependent, while the breakage distribution function is mill and environment independent.

Celik (1988) analyzed breakage rates of anthracite in batch dry and wet grinding. He found that the breakage rate of anthracite showed an acceleration, particularly for long grinding times. To understand this acceleration, 1:1 mixture of coarse (20x30 mesh) anthracite mixed with fine anthracite or fine quartz as a feed for ball milling was tested. Those studies showed that addition of fine particles to the coarse feed increased the breakage rate of the coarse material; also test results revealed that the harder the fine particles the greater the increase in the coarse breakage rate.

Kanda et. al. (1989) ground a 1:1 mixture of coal and iron ore in a laboratory ball mill. Data analysis indicated that there was an increase in the breakage rate of the coal when ground with iron ore compared to when ground alone, while the iron ore breakage rate decreased when ground as a component of a mixture with coal.

Fuerstenau and Abouzeid (1991) investigated the effect of fine particles on the kinetics and energetics of grinding of coarse particles of the same mineral. For this purpose, batch grinding experiments were conducted with dolomite and quartz, the 10x14 mesh size fraction defined as the coarse and -100 mesh as the fine size fraction. The coarse and fine size fractions of the same material were mixed at different coarse to fine ratios (C:F 3:1, 1:1, 1:3, 1:5.7) as a feed to the mill and ground for 1, 2 and 4 min, cumulatively. The results showed that the breakage rate of the coarse fraction increases as the ratio of the fine fraction in the mixture feed increases, while the breakage distribution function remains invariant whether ground alone or in a mixture with the fine fraction. In addition, using the energy split concept they concluded that the specific energy consumed by the coarse fraction increases as the ratio of coarse to fine material in the mill decreases.

Verma and Rajamani (1995) examined the changes in breakage rates for all size intervals as the particle size distribution changed during grinding. Limestone and copper ore were used as test materials, which were fed to the mill separately as mono-sized (10x14 mesh), naturally sized (-10 mesh; 15-20% material in top size interval) distributions and coarse sized (-10 mesh; 30-35% material in the top size interval) samples. Their experiments indicated that the breakage rates varied during grinding so that one of the tests conducted on natural sized limestone showed breakage rates firstly accelerated, then decelerated and accelerated again as the grinding time increased. However, they concluded that, in general, breakage rates of coarse particles increases in the presence of fine particles.

Hoşten and Avşar (1998) investigated the ball-mill grinding kinetics of monosized (14x20 mesh) feed samples of clinker and trass when they were ground individually or as a component in their binary mixtures at two different volumetric proportions (80% clinker + 20% trass and 65% clinker + 35% trass).The breakage rate of clinker (the hard component) did not change significantly when ground alone or in the mixture while the breakage rate of trass (the soft component) decelerated to a small but sensible extent when it was present in a small amount (20% v/v) in the mixture. This rate-deceleration effect was attributed to the lesser probability for trass particles to get caught between grinding balls at such a low proportion in the mixture, which might have also caused a change in the breakage mechanism for the trass particles from shattering to chipping and abrasion. The study also showed that the breakage distribution of clinker remained the same whether it was ground alone or in mixture but that of trass was dependent on the mixture composition, and more trass fines were generated with decreased proportion of trass in the mixture feed.

## 2.7 Concluding Remarks

There are several published research papers focused on the grinding behavior of minerals when ground alone or in a mixture in ball mills. Most of the previous research work was limited to coarse-fine mixtures of the same mineral. In industrial operations, ores containing minerals of varying hardness are ground, which may lead to an abundance of soft mineral fines in the mill charge. The mill charge then will become a mixture of hard-coarse particles and soft-fine particles. The existing literature lacks a detailed, systematic study in this respect. This thesis work, therefore, attempts to reveal the effect of soft fine particles on the grinding kinetics of hard coarse particles.

# CHAPTER 3

# EXPERIMENTAL MATERIALS AND METHODS

## 3.1 Materials

Batch grinding experiments were carried out with quartz and calcite. These two minerals have very close densities (quartz: 2.68 g/cm$^3$, calcite: 2.70 g/cm$^3$), but different degrees of hardnesses on the Mohs scale (quartz: 7, calcite: 3). Quartz sample was acquired as pure quartzite (metamorphosed quartz) from a freshly opened gold mine located at Balikesir in the western part of Turkey. It was intentionally gathered from the waste part of the pit to obtain clear quartzite and prevent diversity in grinding behavior. Calcite sample (98.5% $CaCO_3$, 1.5% $MgCO_3$) was supplied by Omya mining from Kırşehir, which is a quarry mine located in the central part of Turkey.

The as-received quartz and calcite samples weighed about 150 kg and 100 kg, respectively. Both materials were obtained as large chunks and broken down sequentially with laboratory jaw crushers and a roll crusher. The crushed products were firstly screened with a gyratory screen to separate the intended sizes. The materials were then carefully sieved to obtain the grinding test samples of -1.18+0.85 mm size fraction for quartz and -106 µm fraction for calcite and quartz (sieve data given in Figure 3.1). A narrowly sized -150+106 µm calcite and quartz samples were also prepared for the single size tests and to use as a fine component in mixture with coarse component.

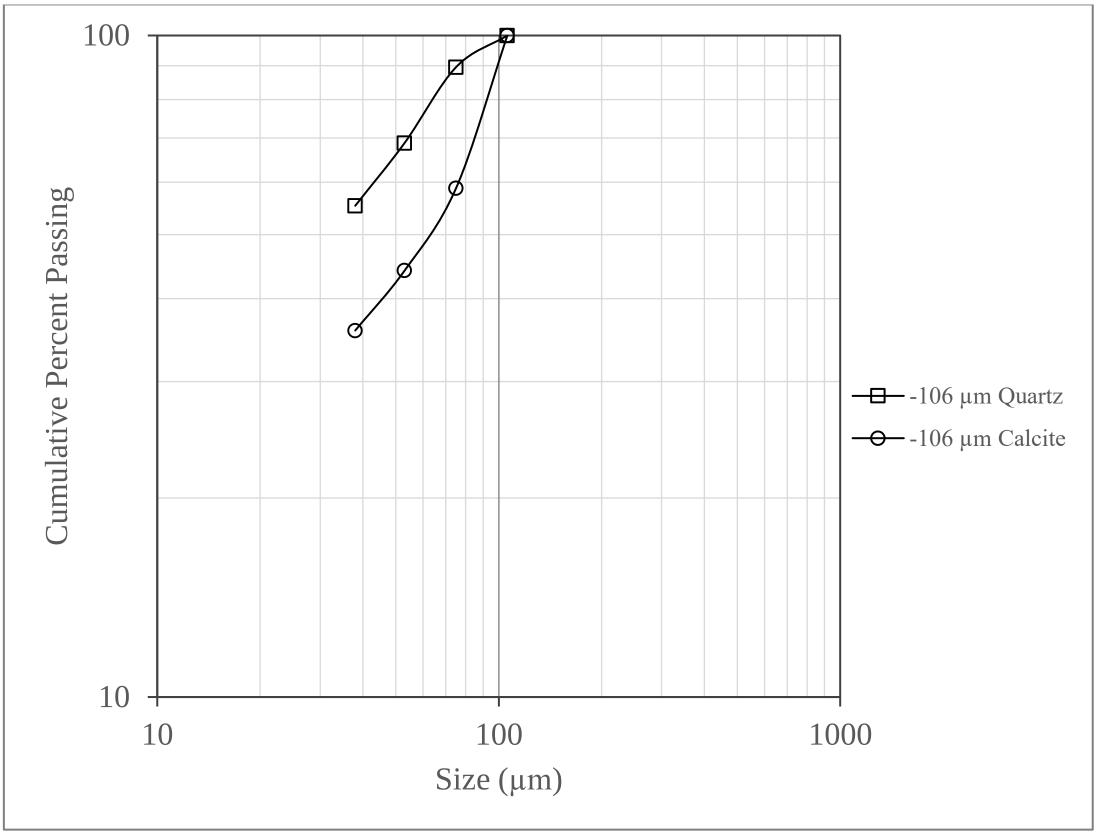


Figure 3.1 Size distributions of -106 µm quartz and calcite

## 3.2 Methods

Batch grinding experiments were carried out in an 18.8 cm diameter by 17.7 cm long laboratory-scale ball mill having four equally-spaced rectangular lifters. The experimental mill and material conditions are tabulated in Table 3.1. One inch (25.4 mm) steel balls were used as the grinding media. A ball load of 8.02 kg were charged which is occupied 35% of the struck volume of the mill assuming ball bed porosity is 0.4. During all experiments, the operational speed of the mill was 60 rpm which is 57% of the critical speed. The material charges were kept constant at 1.00 kg for all tests, which corresponds to 92% of the interstitial space between the balls at rest. In wet grinding experiments, water was added so as to have 60% by weight solids in the pulp.

Table 3.1 Mill characteristics and experimental conditions

| | | |
|---|---|---|
| Mill | | |
| $D$ | Internal diameter (m) | 0.188 |
| $L$ | Length (m) | 0.177 |
| $N_c$ | Critical speed (rpm) | 105 |
| | Operating speed (rpm) | 60 |
| $\phi_c$ | Critical speed ratio (%) | 57 |
| Grinding Media | | |
| $d_B$ | Ball Size (mm) | 25.4 |
| $\rho_{ball}$ | Density of ball (g/cm$^3$) | 7.78 |
| $\phi_B$ | Volume fraction of ball bed in the empty mill | 0.35 |
| $\varepsilon_{ball}$ | Ball bed porosity fraction | 0.40 |
| $M_{ball}$ | Mass of balls (kg) | 8.02 |
| Material | | |
| $\phi_M$ | Volume fraction of particles in ball porosity | 0.92 |
| $\varepsilon_{powder}$ | Material bed porosity fraction | 0.40 |
| $f_c$ | Volume fraction of particle bed in the empty mill | 0.13 |
| Material Density | | |
| | Quartz (g/cm$^3$) | 2.70 |
| | Calcite (g/cm$^3$) | 2.68 |
| $M_{powder}$ | Mass of powder (kg) | 1.00 |
| Wet Grinding | | |
| Pulp density (% solids by weight) | | 60 |

The individually prepared single size coarse quartz (-1.18+0.85 mm) and fine calcite (-106 μm) were mixed at different coarse-to-fine (C:F) mass ratios of 1:0, 3:1, 1:1, 1:3, 1:5.7 as feed to the mill. Before the mill run, the material and the ball load were charged into the mill layer by layer. Then, the mill was sealed and started to rotate using a roller table for given set of time. The grinding times were 0.5, 1, 2, 3 and 4 min, cumulatively. After each grinding time, the mill was stopped and emptied onto a steel grid to separate the sample from the balls, and the balls were brushed to remove any fine material stuck on. The product was split with a riffle into four equal-weight samples, each having about 250-g weight. One of the samples was randomly selected for size analysis, and first underwent wet screening to remove fine (-75 μm) material to overcome screen blinding. The remaining material and -75 µm fraction were dried in an oven and weighed. After that, +75 µm material was sieved dry with a Rotap shaker having a set of sieves down to -106 µm progressing in √2 order. After sieve analyses, the sampled material together with the rest of the mill product were put back into the mill for further grinding. The same procedure was repeated for each grinding time in sequence.

A similar set of experiments was completed replacing the fine calcite (-106 µm) with fine quartz (-106 µm) for comparison purposes. Moreover, single-size grinding experiments conducted with -150+106 µm fraction of calcite and quartz in order to compare with breakage rate of coarse quartz. Furthermore, selected experiments for coarse quartz/fine calcite with different coarse-to-fine ratios (C:F 1:0, 1:1, 1:5.7) were performed by wet grinding having 60% solids by weight. In addition, mixture tests were conducted with narrowly sized (-150+106 µm) calcite or quartz as a fine component at two coarse-to-fine ratios (C:F 1:3, 1:5.7). The data obtained by sieve analysis for one minute was used to estimate the breakage distribution function. Direct determination using the BII method was utilized as proposed by Austin and Luckie, 1972 (Austin et al., 1984) based on the solution of the batch grinding equation. The breakage rate function was determined from the top size fraction remaining. Since all the experiments were completed under identical mill conditions, the power drawn by the mill was kept constant. This enabled determination of the fraction of energy consumed by mineral constitutes in the mill for different mineral mixtures using the energy split consideration developed by Kapur and Fuerstenau (1988).

# CHAPTER 4

# RESULTS AND DISCUSSION

## 4.1 Evaluation of Breakage Functions

### 4.1.1 Breakage Rate Function

Breakage rate parameters of the top size fraction were determined from the size-discretized batch-grinding equation, which has been explained in section 2.4.1. The semi-log plot of coarse fraction remaining as a function of time gives a linear line if the grinding kinetics follow the first-order grinding hypothesis, meaning that the breakage rate does not depend on time. The breakage rate parameters can be obtained from the slope of this line.

Figure 4.1 shows the disappearance plots for dry grinding of -1.18+0.85 mm coarse quartz when mixed with -106 µm quartz fines at different coarse-to-fine ratios of 1:0, 3:1, 1:1, 1:3, and 1:5.7. The linear plots indicate the validity of first-order disappearance kinetics for quartz when ground alone or as a coarse component of coarse/fine mixtures of the same material. It is clearly seen from the plots that the breakage rate (slope of the linear plot) of the coarse fraction increases as the ratio of the fine fraction in the mixture feed increases, particularly above 25% (C:F 3:1). This finding is in agreement with previous work by other researchers (Phatak 1990, Fuerstenau and Abouzeid, 1991, Verma and Rajamani 1995, Fuerstenau et all, 2010).

The breakage rate function variation with the fine material in the feed was plotted in Figure 4.2. As seen from the figure, the breakage rate of the coarse quartz fraction is lowest (0.114 $min^{-1}$) when it is ground alone. Variation in the breakage rate is small when the fine quartz fraction in the feed amounts to 25%, increasing the rate to about

0.121 $min^{-1}$. The breakage rate, however, increases to 0.146 $min^{-1}$ when the feed contains 50% (C:F 1:1) of fine material and to 0.228 $min^{-1}$ for 75% (C:F 1:3) fines in the feed, which is twice the rate when the coarse fraction is ground alone. Finally the breakage rate of the coarse quartz fraction increases to 0.304 min-1 when the fine material comprises 85% (C:F 1:5.7) of the feed. As a conclusion, it can be stated that the breakage rate of the coarse particles increases as the ratio of the coarse material in the feed decreases (or equivalently the ratio of the fine material increases). These results are in close agreement with the findings of previous researchers (Phatak 1990, Fuerstenau and Abouzeid, 1991, Verma and Rajamani 1995, Fuerstenau et all, 2010), and will serve as the baseline for the major purpose of this thesis study, in which the effect of soft fine material on the breakage rate and distribution function of closely sized coarse hard particles was investigated. For this purpose, calcite, having the same density as quartz, was selected as the soft mineral. Thus, -1.18+0.85 mm quartz and -106 μm calcite mixtures at different mass ratios give a combination of the hard-and-coarse material and the soft-and-fine material.

Figure 4.3 shows the disappearance plots for dry grinding of -1.18+0.85 mm coarse quartz when mixed with -106 μm calcite fines at different coarse-to-fine ratios of 1:0, 3:1, 1:1, 1:3, and 1:5.7. This figure shows the same trend for the quartz-calcite mixtures as it was indicated by the quartz-quartz mixtures presented in Figure 4.1, that is, there is again a significant increase in the breakage rate of the coarse hard component when the percentage of the fine soft component exceeds 50% (C:F 1:1). However, it is clearly seen from Figure 4.4 that the soft fine component is not as effective as the hard fine component in increasing the breakage rate of the coarse particles with increasing proportion of the fines in the mixture. When the fine material comprises 85% (C:F 1:5.7) of the feed, the breakage rate of the hard coarse component is reduced from 0.304 $min^{-1}$ to 0.253 $min^{-1}$ if the hard fine component (quartz) is replaced by the soft fine component (calcite).

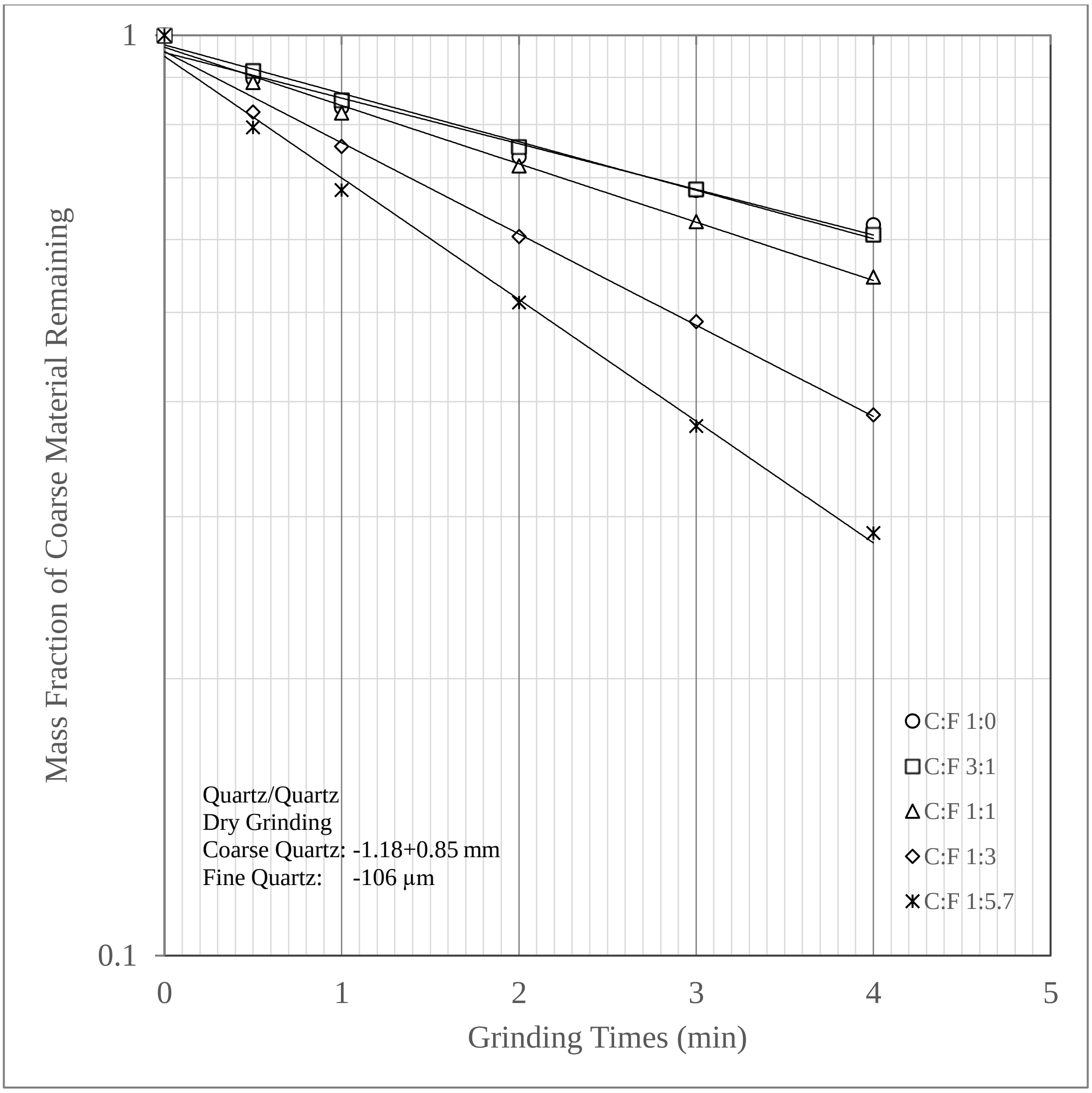


Figure 4.1 The first-order disappearance plots for dry grinding of coarse (-1.18+0.85 mm) quartz when mixed with fine quartz (-106 µm) at different mass ratios.

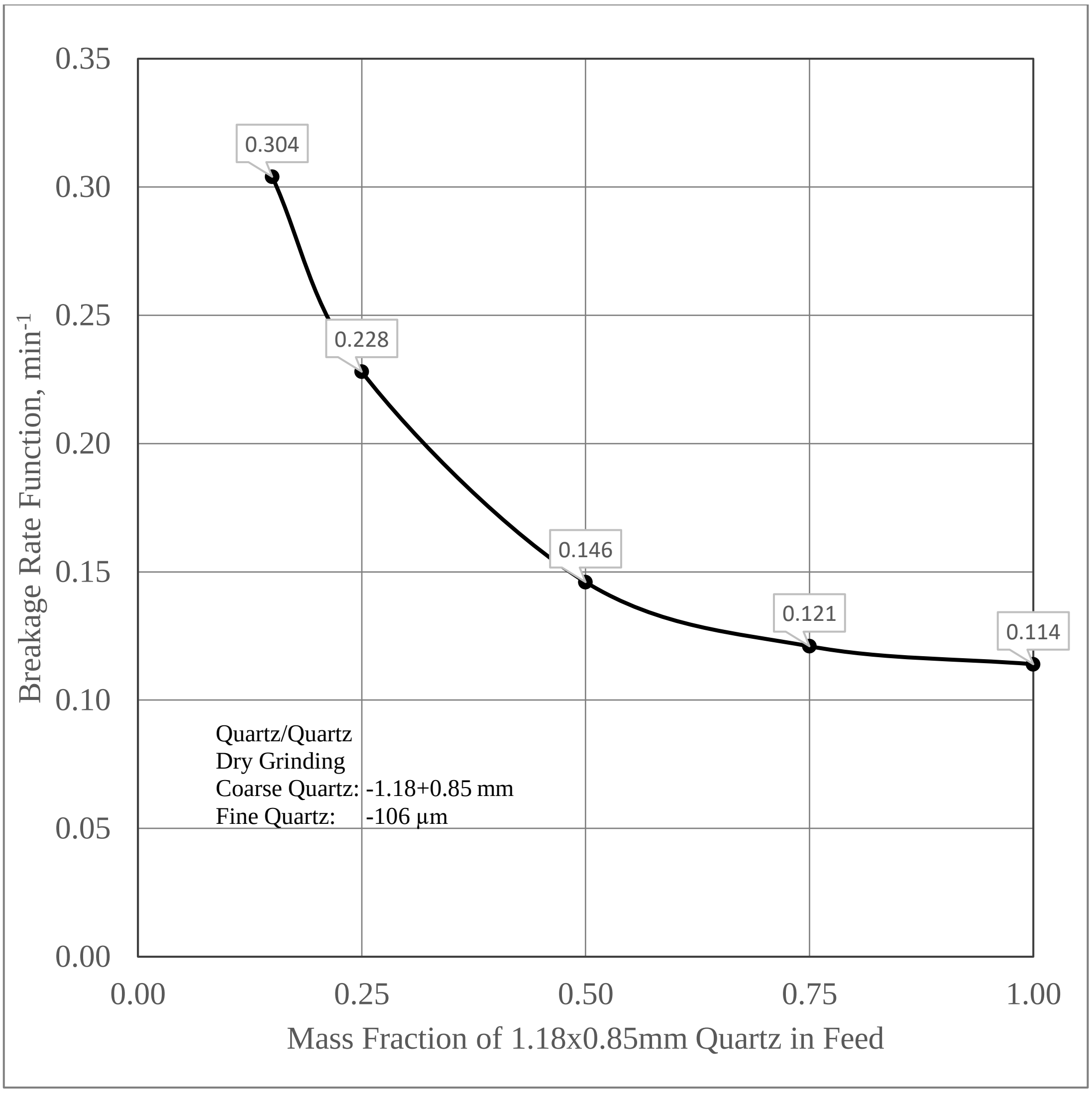


Figure 4.2 Breakage rate function plot of quartz (-1.18+0.85 mm) as a function of its mass fraction when mixed with fine (-106 µm) quartz for dry grinding

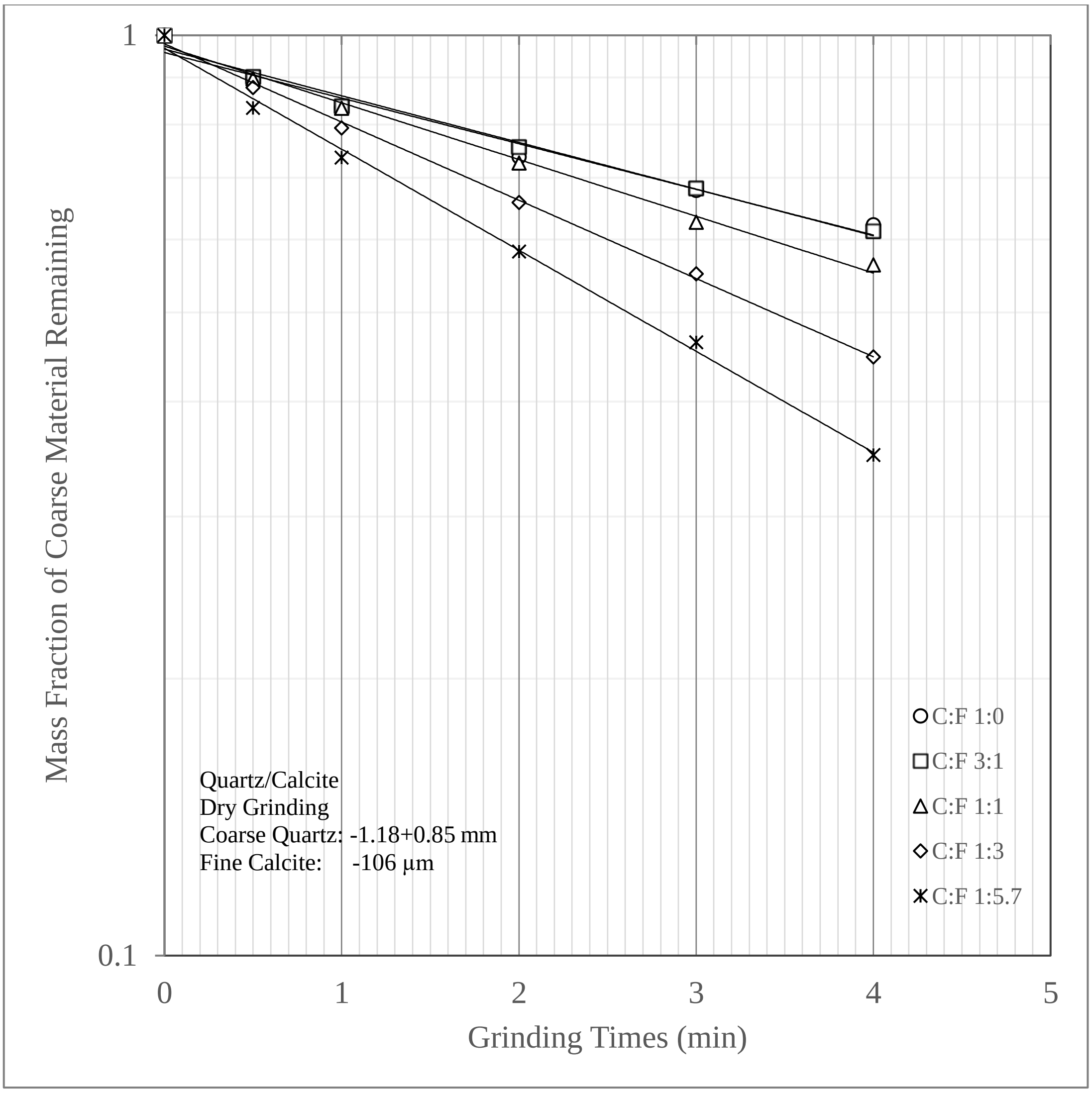


Figure 4.3 The first-order disappearance plots for dry grinding of coarse (-1.18+0.85 mm) quartz when mixed with fine calcite (-106 µm) at different mass ratios.

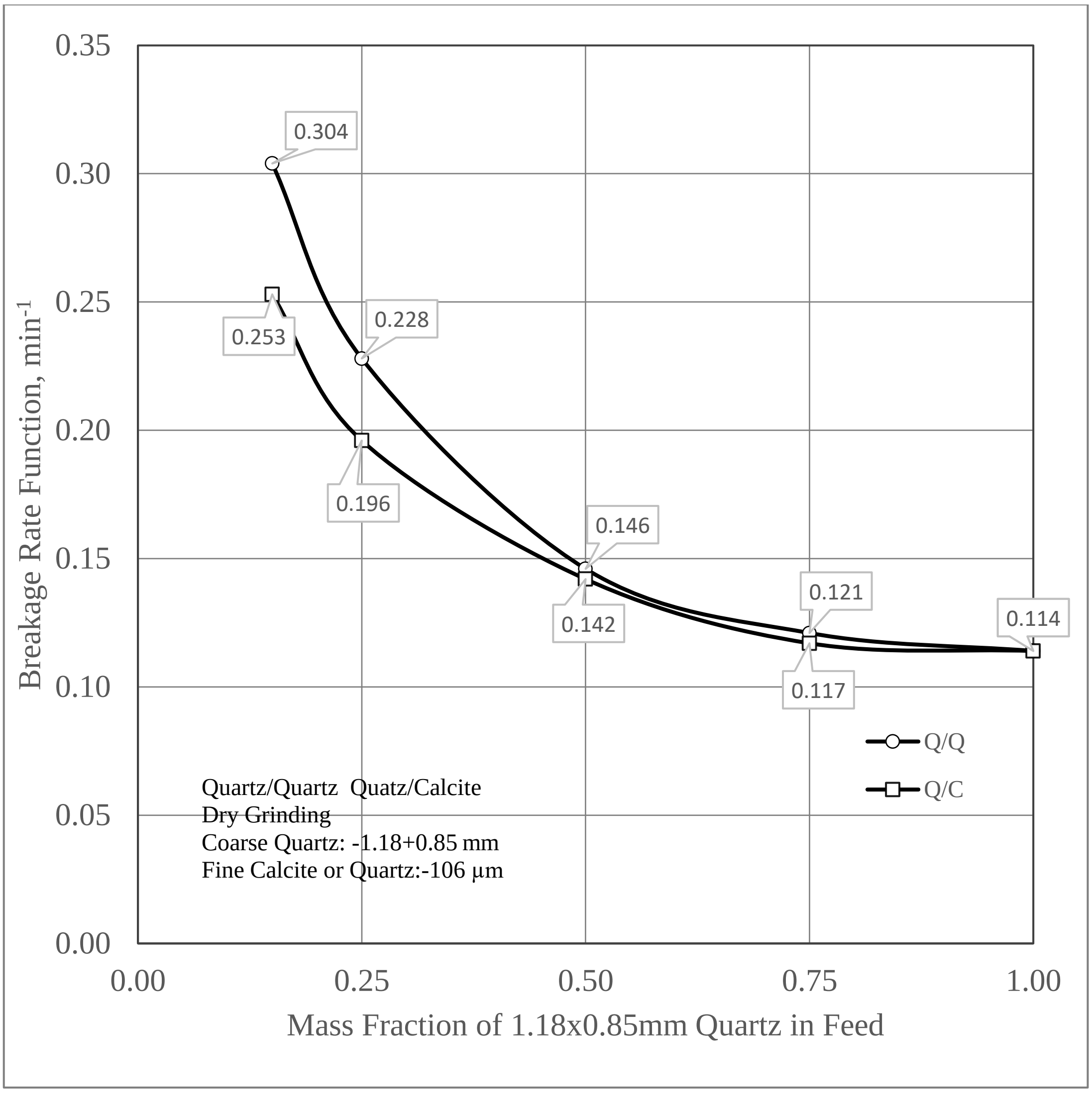


Figure 4.4 Breakage rate function plots of quartz (-1.18+0.85 mm) as a function of its mass fraction when mixed with fine (-106 µm) quartz or fine (-106 µm) calcite for dry grinding.

The increase observed in the breakage rate of the coarse component in the presence of fines was also tested by adding mono-size fine component (-150+106 µm), rather than a wide range of fine sizes (-106 µm), while keeping other operating parameters constant. This set of experiments was expected to delineate the effect of the mineral hardness of the fines independent of the effect that could arise from the difference in the size distributions of the -106 µm quartz and calcite fines. For this purpose, both quartz-quartz and quartz-calcite mixtures were tested at coarse-to-fine ratios of 1:3 and 1:5.7. As shown from Figure 4.5 and Table 4.1, similar to previous test, as the mono-size (-150+106 μm) fine fraction of quartz or calcite increasing in the feed, coarse quartz grinds faster. For the same coarse-to-fine ratios, the magnitude of the increase in the breakage rate of the coarse quartz is again lower when calcite is used as the soft fine component. A plausible cause for this could be less brittle calcite fines absorbs more energy, leaving lesser for the breakage of coarse quartz particles. Figure 4.6 and Figure 4.7 show that the finer the fine component in the mixture is, the more significant is its effect on increasing the rate function of the coarse component.

Table 4.1 Breakage rate functions of coarse quartz when ground alone or in mixture with fine component (quartz or calcite)

| Grinding Condition | Feed Material Sizes | Mixture C:F Ratios | Coarse Quartz Breakage Rate Function ($min^{-1}$) | |
|---|---|---|---|---|
| | | | w/ Fine Quartz | w/ Fine Calcite |
| DRY | Coarse Quartz: -1.18+0.85 mm Fine Quartz or Calcite: -106 μm | 1:0 | 0.114 | 0.114 |
| | | 3:1 | 0.121 | 0.117 |
| | | 1:1 | 0.146 | 0.142 |
| | | 1:3 | 0.228 | 0.196 |
| | | 1:5.7 | 0.304 | 0.253 |
| WET | Coarse Quartz: -1.18+0.85 mm Fine Quartz or Calcite: -106 μm | 1:0 | NA | 0.130 |
| | | 3:1 | NA | NA |
| | | 1:1 | NA | 0.164 |
| | | 1:3 | NA | NA |
| | | 1:5.7 | NA | 0.311 |
| DRY | Coarse Quartz: -1.18+0.85 mm Fine Quartz or Calcite: -150+106 μm | 1:0 | NA | NA |
| | | 3:1 | NA | NA |
| | | 1:1 | NA | NA |
| | | 1:3 | 0.139 | 0.125 |
| | | 1:5.7 | 0.162 | 0.149 |

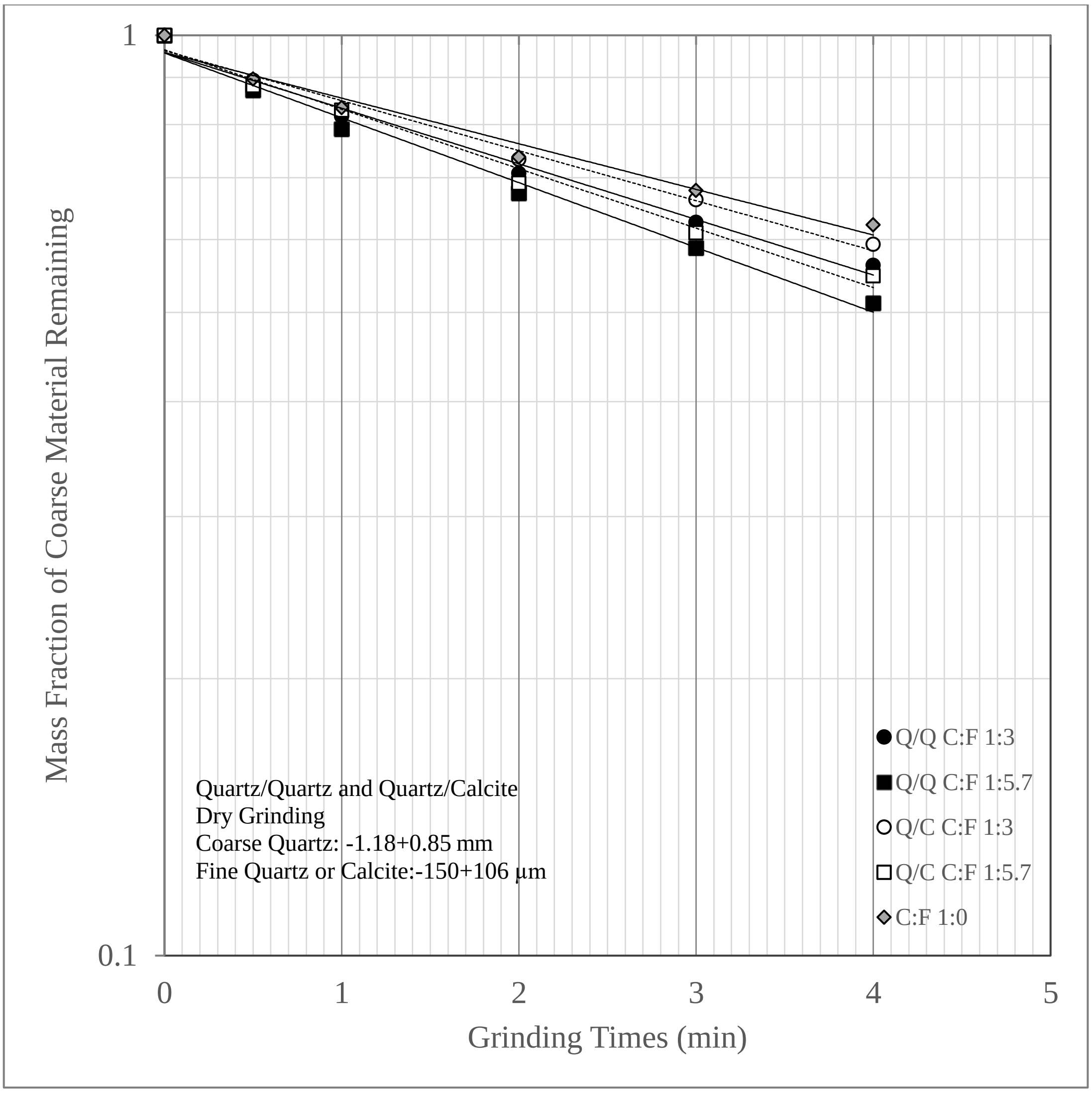


Figure 4.5 The first-order disappearance plot for dry grinding of coarse (-1.18+0.85 mm) quartz as single or mixed with mono-size fine fractions (-150+106 µm) of quartz or calcite at different mass ratios.

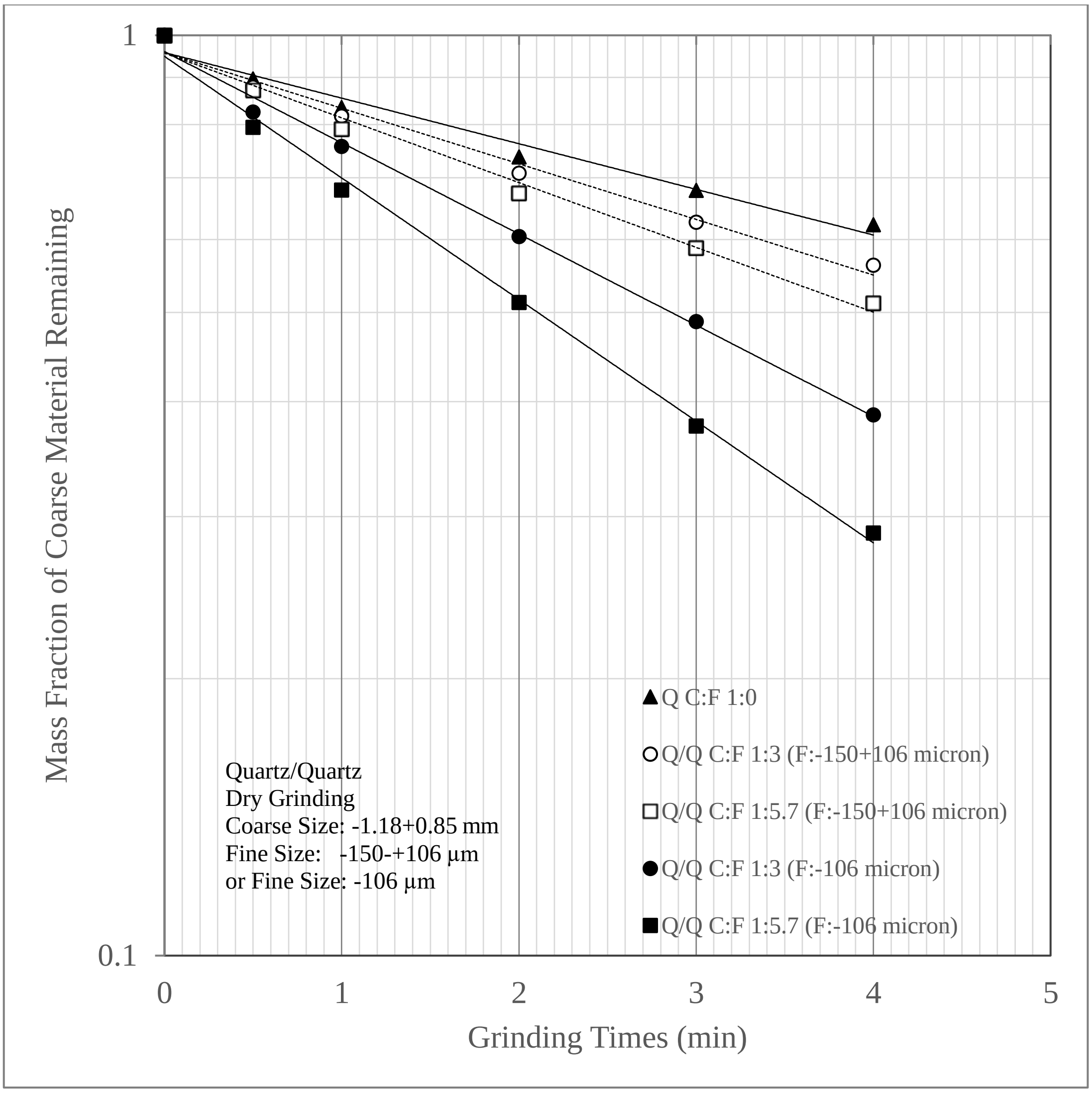


Figure 4.6 The first-order disappearance plot for dry grinding of coarse (-1.18+0.85 mm) quartz as single or with addition of wide range fine (-106 μm) or mono-size fine fraction (-150+106 μm) of quartz at different mass ratios.

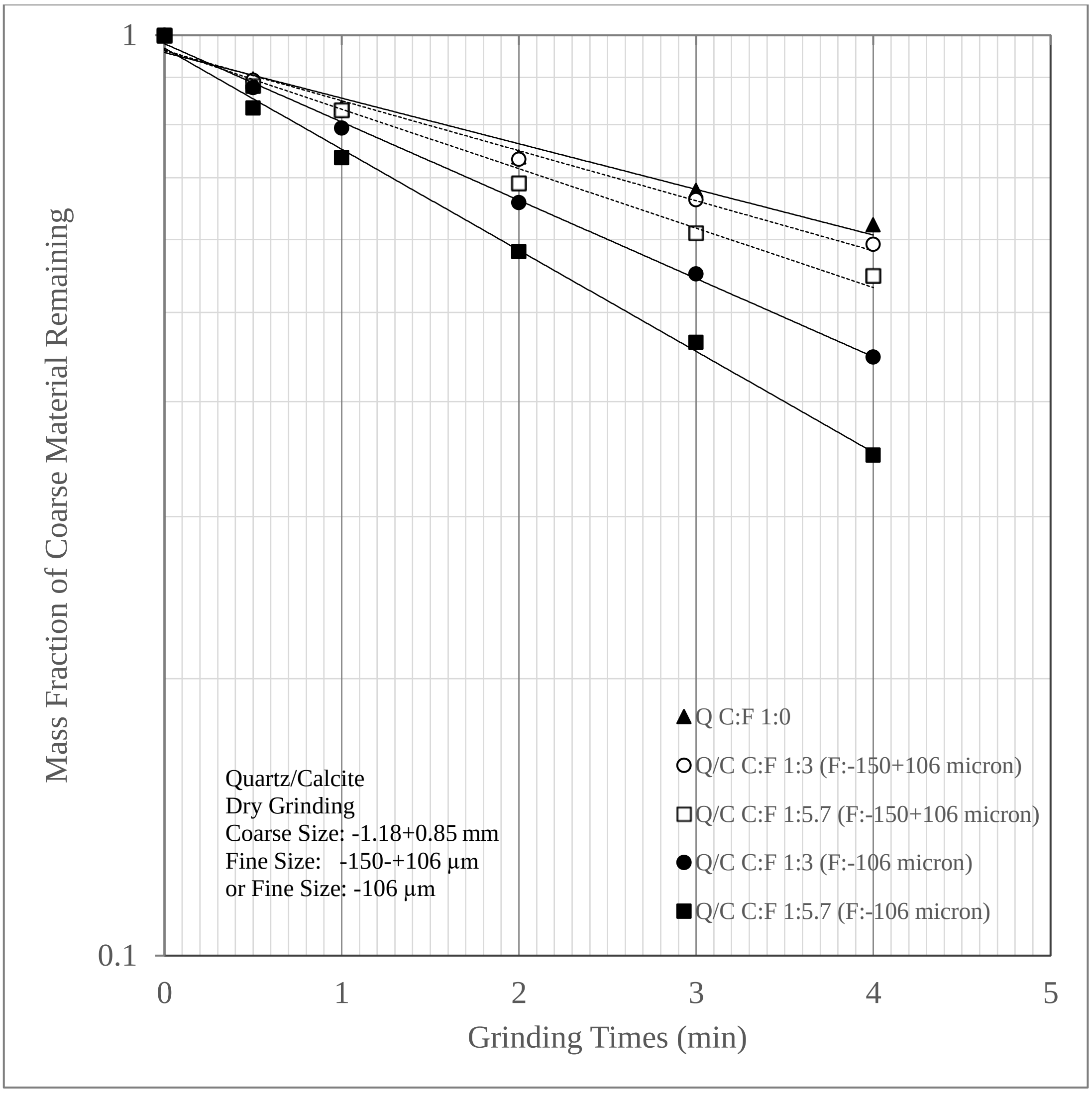


Figure 4.7 The first-order disappearance plot for dry grinding of coarse (-1.18+0.85 mm) quartz singly or with the addition of wide range fine (-106 µm) or mono-size fine fraction (-150+106 µm) of calcite at different mass ratios.

In this study, heterogeneous mixtures in terms of material type and size were fed to the mill. It is well known that fine particles are stronger than coarse particles of the same material, and therefore finer size fractions have lower breakage rate functions (or lower grindability). Considering the same size range, particles of a hard mineral will have a lower breakage rate function than particles of a soft mineral when both are ground alone. In mixture grinding of hard/coarse with soft/fine mineral constituents, however, it is reasonable to expect that we would have different combinations of the relative magnitudes of the breakage rate functions of the constituents in the mixture. Then, it is very likely that the soft/fine mineral component may have a breakage rate function equal to or lower than that of the hard/coarse component in the mixture. In fact, as shown in Figure 4.8, this is the case in this study because -150+106 µm calcite, when ground alone, gives a lower breakage rate function than the coarse size fraction (-1.18+0.85 mm) of the harder mineral quartz. It is a known fact that the breakage rate of the singly ground minerals decreases linearly in the size ranges below about 1 mm. Therefore, it is obvious that the breakage rate functions of the mono-size fractions of calcite in the -106 µm range should be increasingly lower than the breakage rate function of -1.18+0.85 mm size fraction of quartz. In other words, the mineral being softer in the Mohs Scale of mineral hardness behaves as the harder mineral from grindability point of view. Table 4.2 showed mono-size breakage rate function of quartz and calcite for coarse and fine sizes.

Table 4.2 Breakage rate functions of mono-size quartz and calcite

| **Feed Material** | **Feed Size** | **Breakage Rate Function ($min^{-1}$)** |
|---|---|---|
| Coarse Calcite Ground Alone | -1.18+0.85 mm | 0.312 |
| Coarse Quartz Ground Alone | -1.18+0.85 mm | 0.114 |
| Fine Calcite Ground Alone | -150+106 micron | 0.093 |
| Fine Quartz Ground Alone | -150+106 micron | 0.075 |

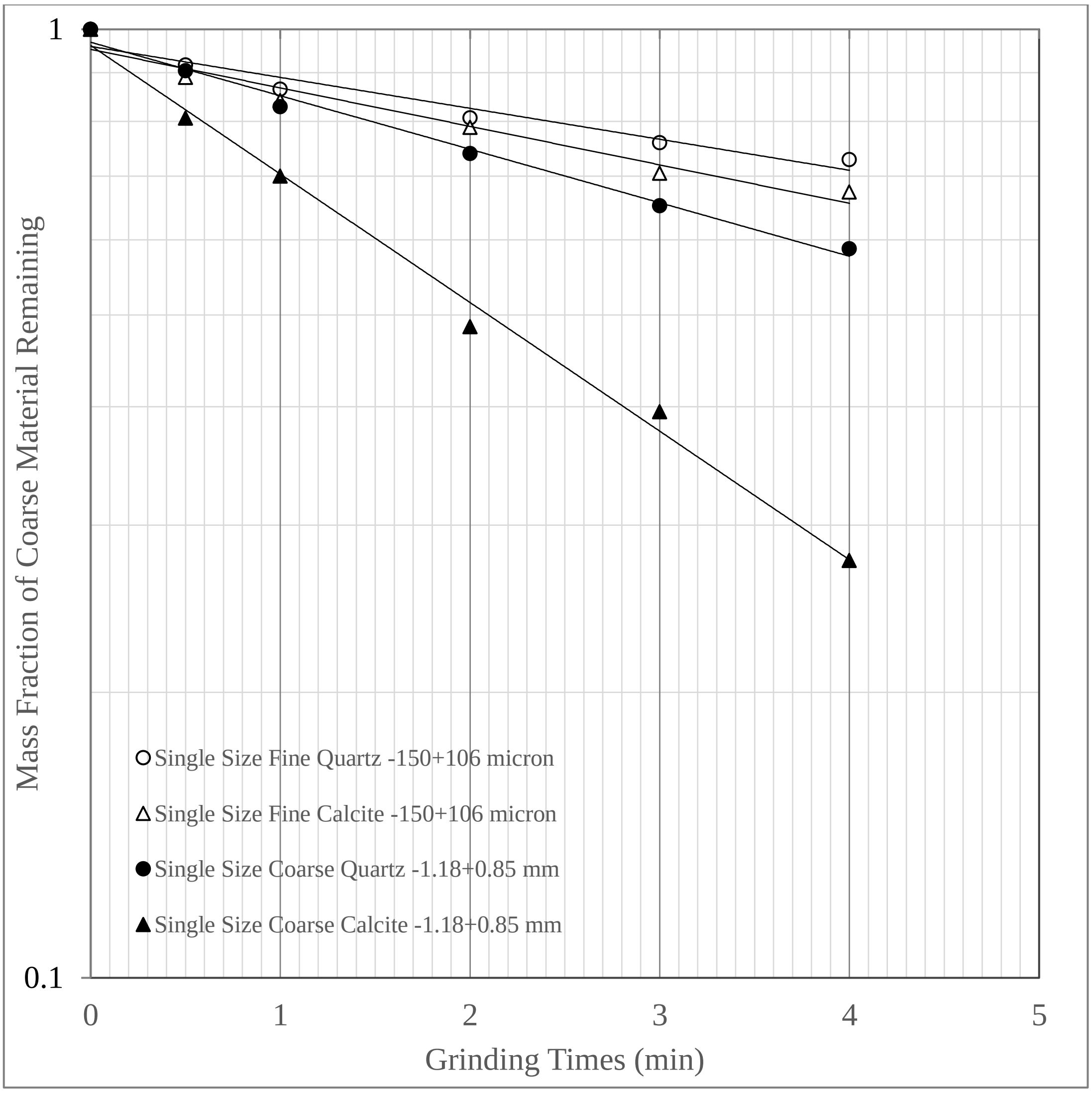


Figure 4.8 The first-order disappearance plots for dry grinding of single size coarse (-1.18+0.85 mm) quartz and calcite, single size fine (-150+106 μm) quartz and calcite, individually.

A plausible reason for the increase in the breakage rate of the coarse component with the addition of fine particles, irrespective of being soft or hard fines, could be the larger size of the coarse particles that make them more easily caught or nipped by grinding media and get broken preferentially. This was also put forward by previous researchers having similar investigation on the coarse and fine mixture of the same material (Phatak, 1990, Fuerstenau et all, 2010). Another reason for this situation could be that a part of the applied energy that was not able to break the fine particles was transmitted to the underlying coarse particles, thus, resulting in more energy consumption by the coarse particles.

As mentioned above, although the soft and the hard fines both caused and increase in the breakage rate function of the coarse hard component, the soft fines were not as effective in this respect, as the hard fines. This phenomenon could be explained with the findings of Venkataraman and Fuerstenau, (1984) and Kanda et. al. (1989). These researchers, ground mixtures of soft and hard materials of the same size. Both investigations revealed that grinding the hard component (quartz or iron ore) with the soft component (calcite or coal) of the same narrow size range lowered breakage rate function of the hard component as compared to the case when it was ground alone. Meanwhile, the breakage rate of the soft component was increased. In other words, softer particles consumed more energy when ground with the hard particles of the same size range. To explain this result from physical point of view Venkataraman and Fuerstenau (1984) noted that if the grinding media nipped soft and hard particles together, softer particles are broken down more easily before the harder material takes necessary impact force to break. Then, for grinding of the mixtures of coarse hard and soft fine minerals, we should expect that two phenomena will be in effect in determining the value of the breakage rate function and partitioning of the input energy for either of the components in the mixture. One is their relative probability of being nipped by the grinding balls, which is dependent on the relative particle sizes of the hard and soft components and also their proportions in the mixture. The second phenomenon has to do with the relative magnitudes of the energy absorbed by the mixture components before their fracture, which is also a function of the particle size as well as the material properties.

### 4.1.2 Breakage Distribution Function

In any mill operation, different breakage mechanisms, namely, fracture, chipping, and abrasion, are active to break the material. The breakage distribution function depends on these breakage mechanisms (Austin et al., 1984). If any breakage mechanism dominates the other ones, the breakage distribution will be affected because each of the mechanisms yields a different type of product. For example, if chipping and abrasion dominates in the mill, more fines will be generated in the product. The breakage distribution function allows analysis of the product data in a phenomenological manner.

Figure 4.9 and Figure 4.10 presents the plots of the cumulative breakage distribution function versus normalized size for the coarse quartz fraction (-1.18+0.85 mm) when ground in a mixture with fine quartz (-106 μm) and fine calcite (-106 μm), respectively. Breakage distributions are normalizable for all coarse-to-fine ratios. As seen from the plots, the breakage distribution function does not vary even though the amount of fines increases in the mixtures. For comparison purposes, cumulative breakage distribution functions of coarse quartz/fine quartz and coarse quartz/fine calcite mixtures are given together in Figure 4.11. It is seen from the figure that the cumulative breakage distribution function of the coarse quartz remains unchanged irrespective of the mixture of fine quartz or fine calcite, where both of the tests were conducted under identical mill operation conditions.

Since the breakage distribution function remains invariant in mixtures, it can be concluded that the breakage distribution function is environment independent. This finding conforms quite well to the observation reported by previous researchers who tested different mineral mixtures of the same size or different sizes of same material (Venkataraman and Fuerstenau, 1984, Phatak, 1990, Fuerstenau and Abouzeid, 1991).

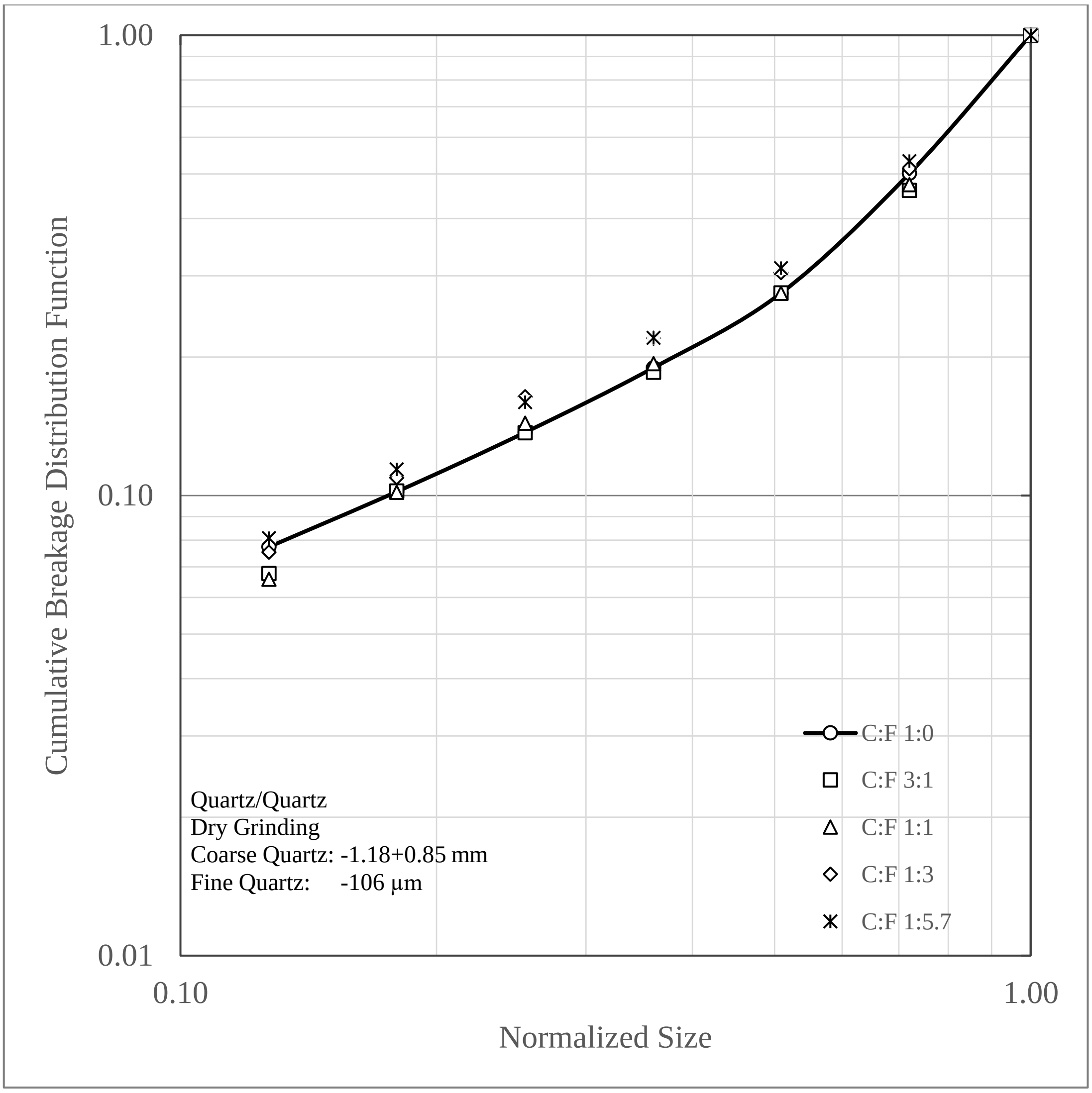


Figure 4.9 A plot of the cumulative breakage distribution function of dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and fine quartz (-106 µm) at different mass ratios.

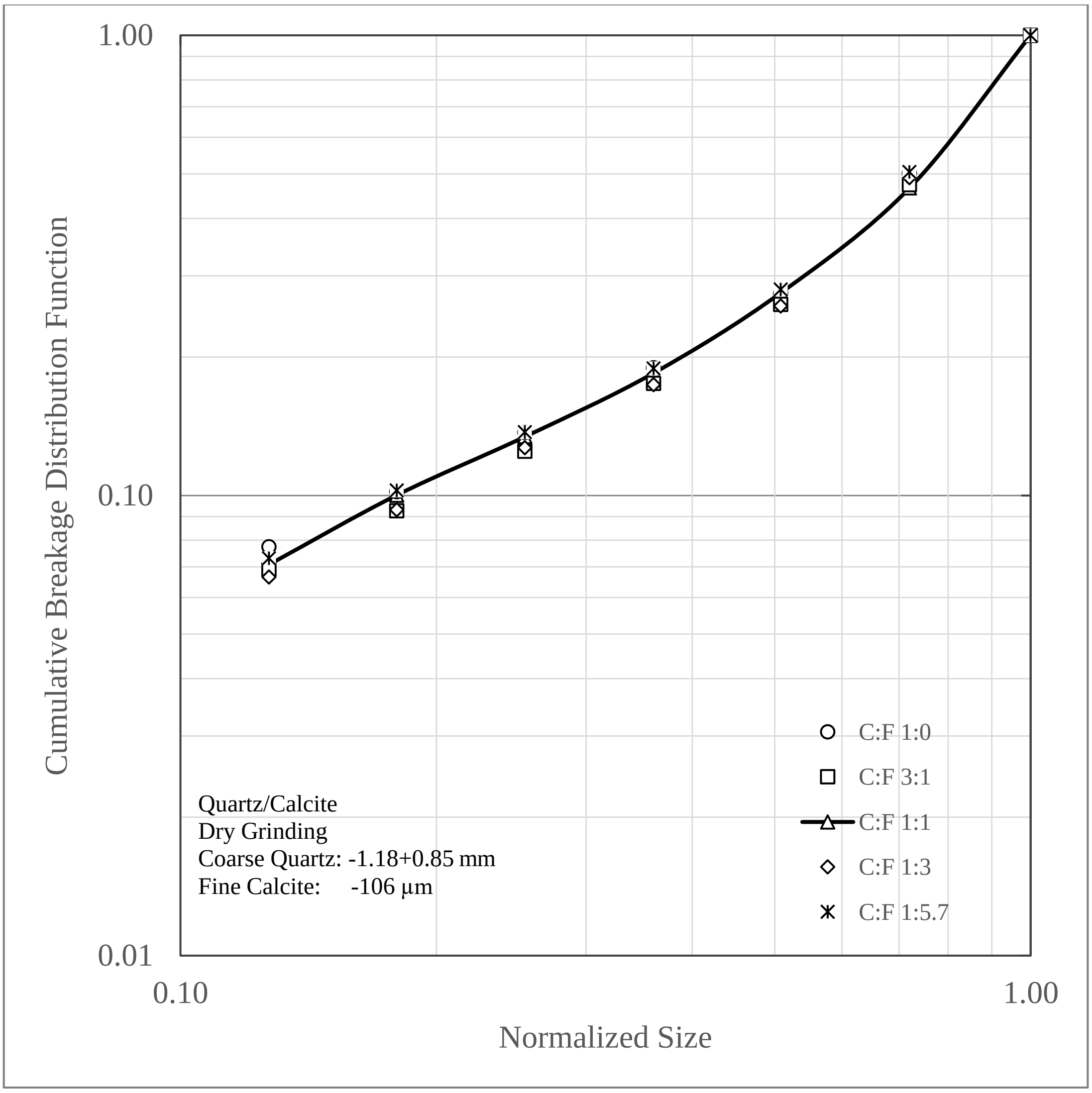


Figure 4.10 A plot of the cumulative breakage distribution function of dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and fine calcite (-106 µm) at different mass ratios.

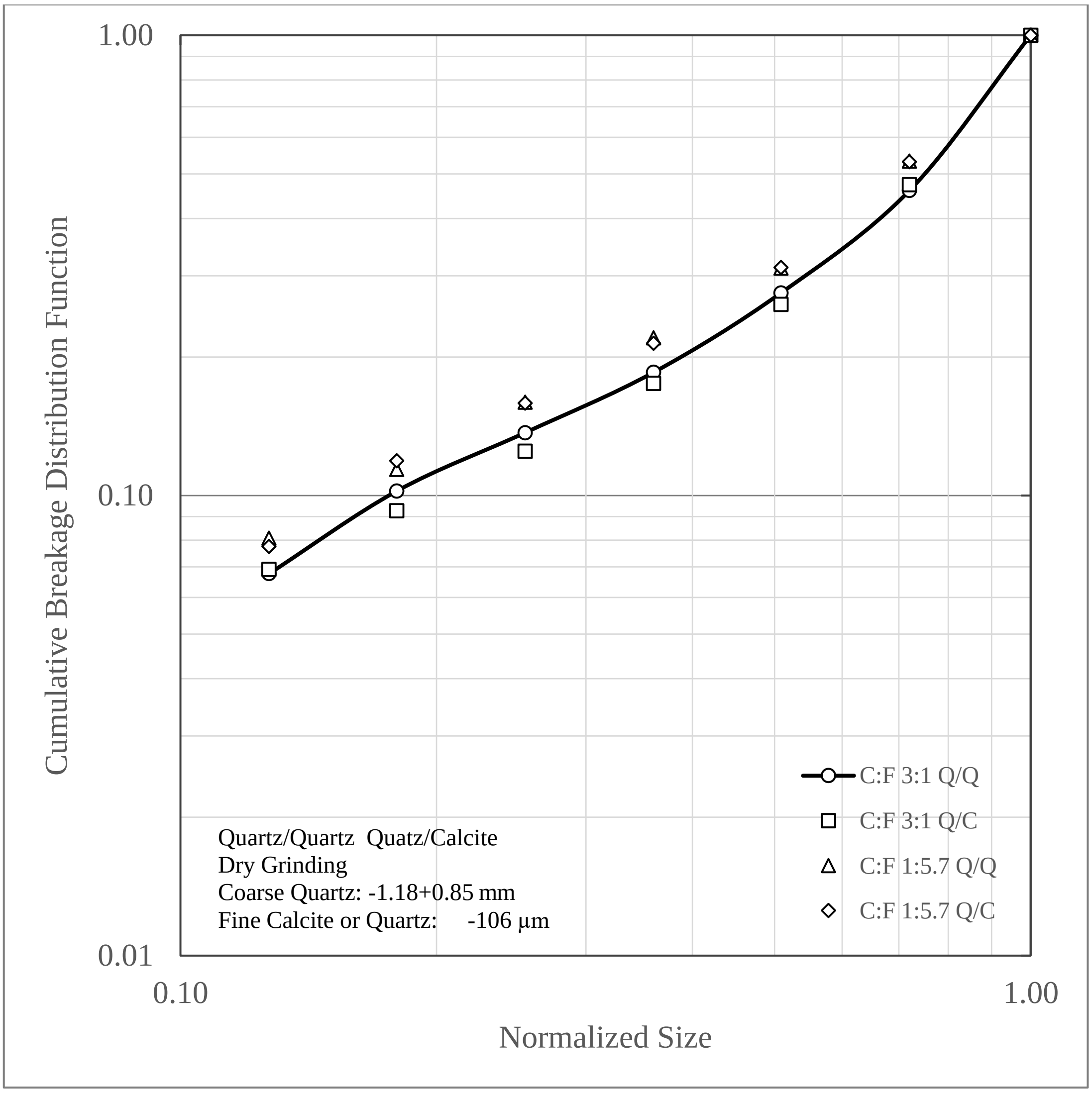


Figure 4.11 A plot of the cumulative breakage distribution function of dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and fine quartz or fine calcite (-106 µm) at different mass ratios.

## 4.2 Wet Grinding Tests

In order to confirm the results attained by dry grinding of coarse-quartz/fine-calcite mixtures, wet grinding experiments were also performed. Three different coarse to fine-ratio mixtures (C:F 1:0, 1:1, 1:5.7) were chosen for the wet grinding experiments, which were conducted at 60% solids by weight and other variables held constant as in dry grinding. It is commonly observed that wet grinding is more efficient than dry grinding (Austin et al., 1981; Tangsathitkulchai, 2002). Austin et al. (1984) postulate that grinding of the materials in the presence of water enhances the grinding rate, and that the enhancement could result from the water enabling the grinding media to transmit the mechanical actions to the particles better causing acceleration of the breakage rate. On the other hand, the primary progeny distribution approximately remains constant.

The disappearance plots for the wet grinding experiments are shown in Figure 4.12. For comparison, dry grinding results of same experimental conditions (mixtures) are also given in the same figure. Similar to the dry grinding case, in wet grinding, breakage of hard coarse particles occurs more rapidly in the presence of fine material, and as the fraction of the fine particles increases in the mixture the breakage rate increases as well. Additionally, the marked deviations of wet grinding results compared to the dry grinding results for all compositions tested confirm that wet grinding leads to higher rates of breakage. This must result from the known fact that fine material suspended in the liquid is removed from the grinding zone, leaving mostly the coarse particles for breakage (Fuerstenau and Abouzeid, 1991).

Figure 4.13 presents the variations of the breakage rate functions of the hard coarse component in both wet and dry grinding for the same data set. As seen from the figure, the increase in the breakage rate of the wet grinding experiments with the addition of soft fines follows the same trend as indicated by dry mixture grinding, meaning that the wet grinding test also confirms the finding that the addition of soft fine particles to the hard coarse particles increases the breakage rates of the coarse particles.

The cumulative breakage distribution functions of the wet grinding experiments together with dry grinding for the same data set are shown in Figure 4.14. Wet grinding result show that breakage distribution are self-similar or normalizable for different fine-to-coarse ratios in hard coarse/soft fine mixtures. Besides, it seems that grinding of hard coarse particles in the presence of soft fine material, irrespective of wet or dry conditions, results in the same type of breakage product distribution.

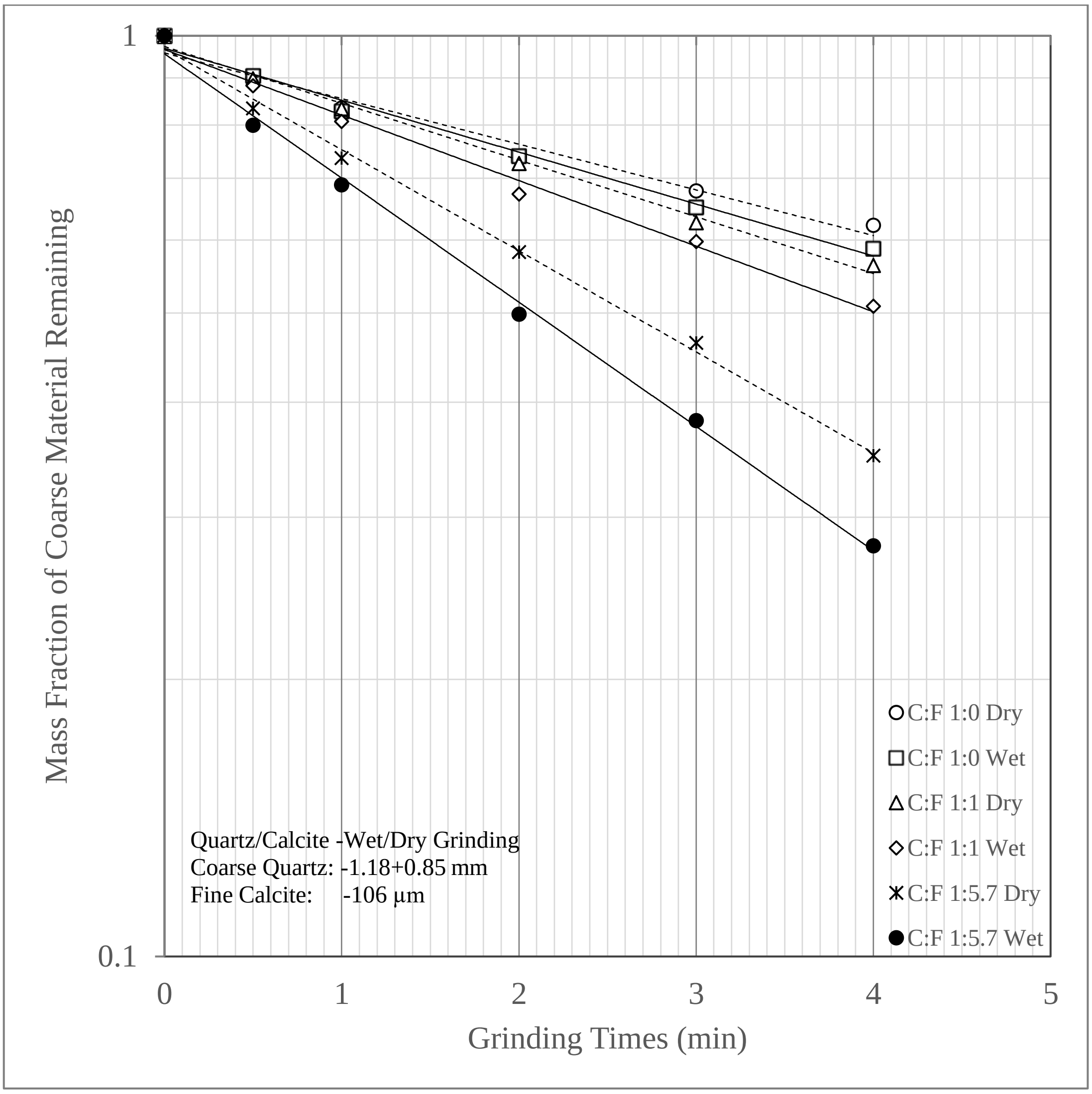


Figure 4.12 The first order disappearance plots for dry and wet grinding of coarse quartz (-1.18+0.85 mm) when mixed with fine (-106 µm) calcite at different mass ratios.

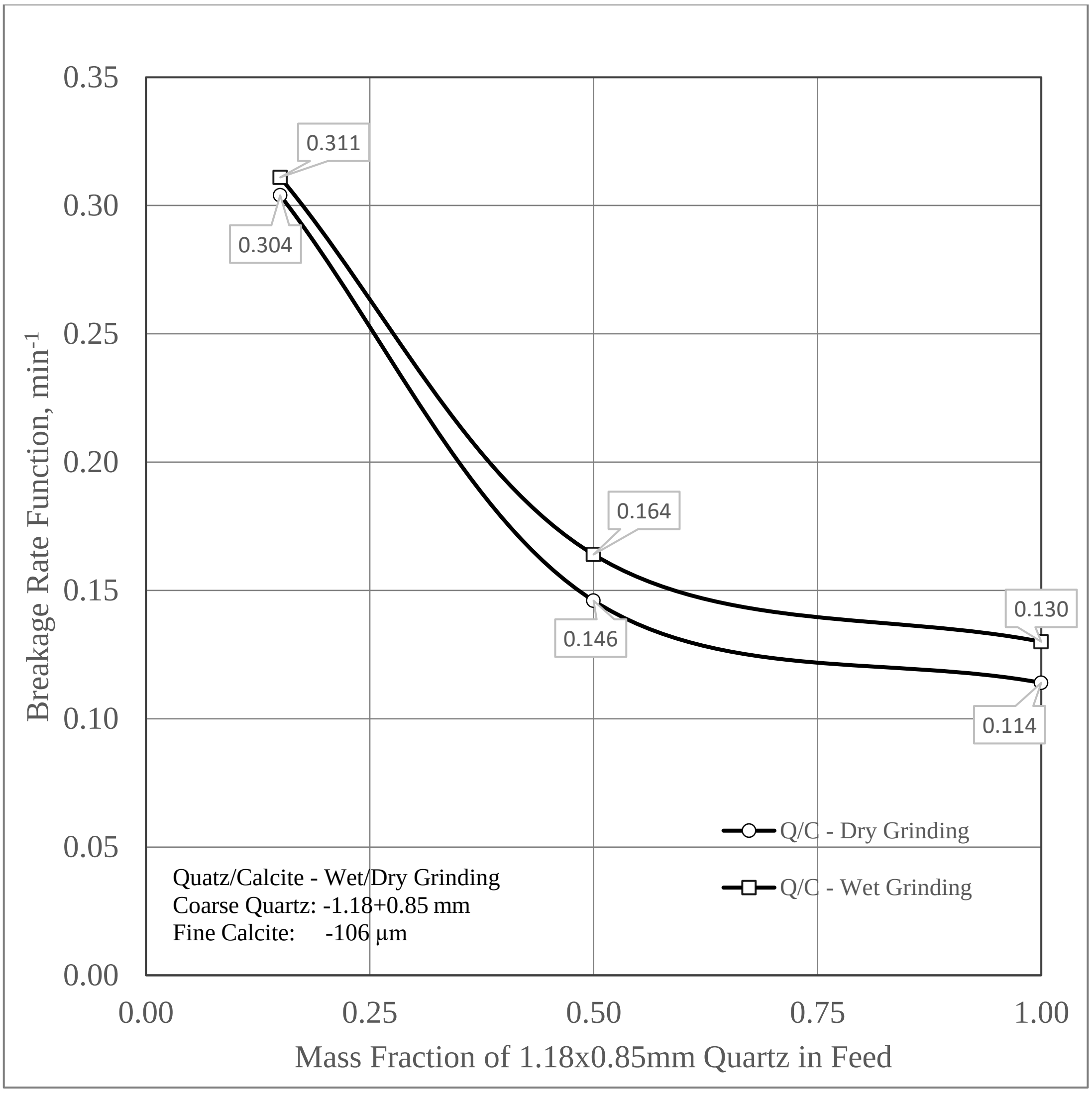


Figure 4.13 Breakage rate function plots of coarse (-1.18+0.85 mm) quartz as a function of its mass fraction when mixed with fine (-106 µm) calcite for wet and dry grinding.

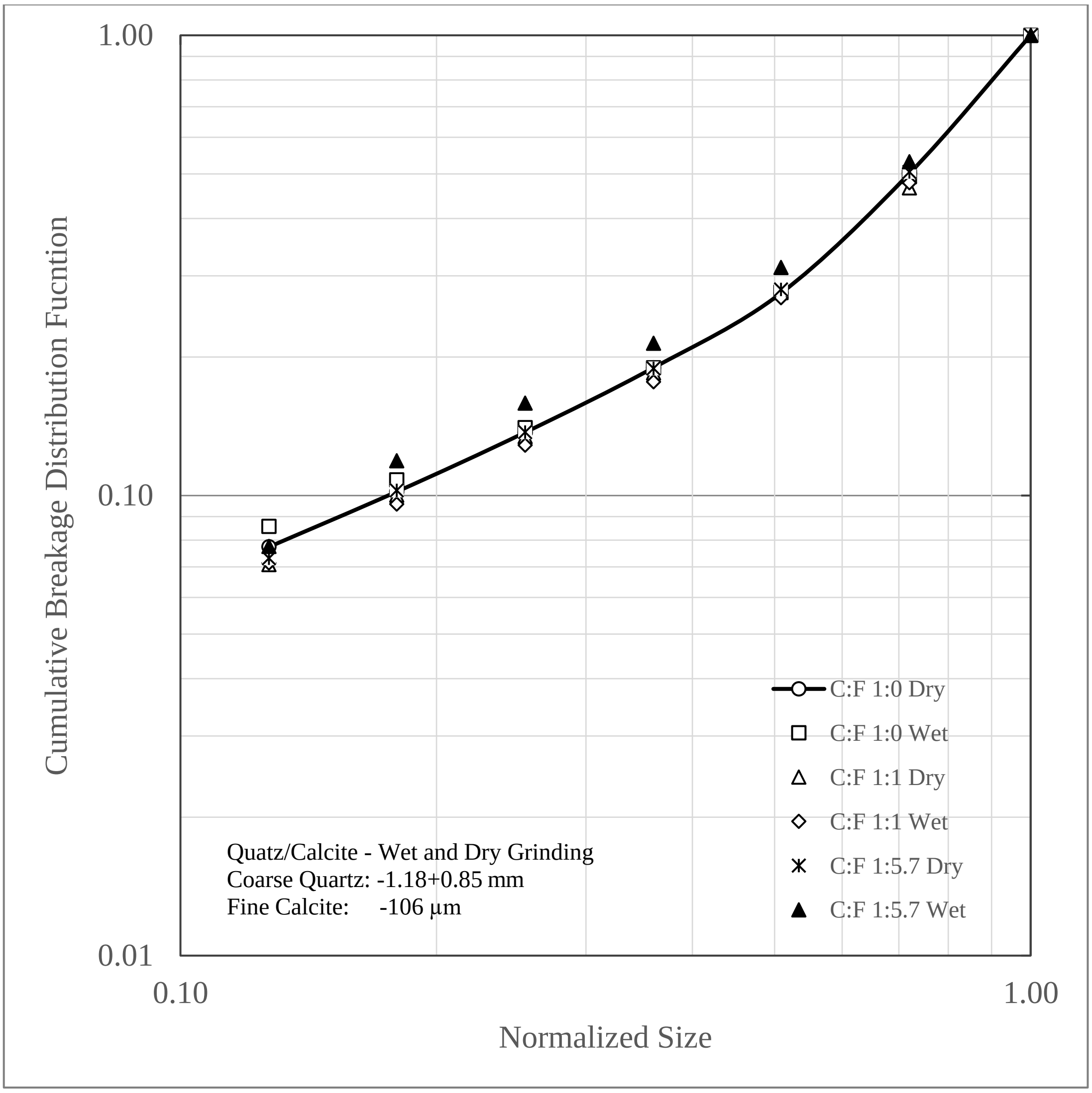


Figure 4.14 A plot of the cumulative breakage distribution function of wet grinding of mixture of coarse quartz (-1.18+0.85 mm) and fine calcite (-106 µm) at different mass ratios.

## 4.3 Energy Split Consideration

Because all batch dry grinding experiments were carried out under identical mill and operational conditions (same fractional filling by media and material, and same critical rotational speed) the power draft and specific grinding energy would be almost the same, being independent of the grinding environment. From the experimental results, it is observed that the hard coarse component grinds faster in the mixture with fine hard particles compared to the mixture with fine soft particles. This might be a result of material effects of fine particles so that soft fine particles consume more specific grinding energy compared to hard fine particles in mixture with coarse hard particles.

In order to test this, the energy split concept developed by Kapur and Fuerstenau (1988) is applied. The approach enables calculation of the distribution of grinding energy among the constituents of minerals using the breakage kinetics. This concept is defined as the ratio of energies expended per unit mass of a material when ground in a mixture environment and ground alone for the same time interval. The energy split factor of the coarse component can be found with the following formula:

$$\mathrm{ES_c} = \frac{\mathrm{E_{cm}}}{\mathrm{E_{ca}}} \tag{26}$$

where $\mathrm{ES_c}$ is the energy split factor of the coarse component, $\mathrm{E_{ca}}$ and $\mathrm{E_{cm}}$ and are the energy expended per unit mass (specific energy) of coarse material when ground alone and in a mixture, respectively. The energy split factor can be computed in terms of breakage rate functions with the following formula:

$$\mathrm{ES_c} = \frac{\ln(m_{cm}(0)/m_{cm}(t))}{\ln(m_{ca}(0)/m_{ca}(t))} \tag{27}$$

where $m_{cm}(0)$ and $m_{cm}(t)$ are the mass fractions of coarse size material at the beginning and time t, respectively. By using Eq. (27), energy split factors of coarse quartz in a mixture of fine quartz or fine calcite were computed.

Figure 4.15 displays the energy split factors of coarse quartz as a function of its mass fractions when it was ground in a mixture with fine quartz or calcite. It is seen that the energy split factor of the coarse quartz increases as its proportion decreases in the mixture for both fine hard quartz and fine soft calcite. Thus, it can be said that increase in the breakage rate function of coarse quartz with fine addition is a direct result of the increase in the specific energy consumption of the coarse quartz. The results also indicate that the split factor value for a given coarse-to-fine ratio is lower when the fine material in the mill feed is soft calcite rather than hard quartz.

Considering that the specific grinding energy is the same for all test materials, by using the energy split factor, the fractional energy by coarse component were computed which are shown in Figure 4.16. It is observed from this figure that the fraction of energy consumed by coarse quartz in the mixture with fines (hard or soft) deviates from the linear line which is followed if energy is distributed by the components in proportion to their mass fractions in the mixture. Besides, it is clearly seen that the fraction of energy consumption of coarse particles when ground with fine quartz is higher than with fine calcite. Expressed in another way, fine calcite consumes more energy compared to the fine quartz in the similar mixture with coarse quartz.

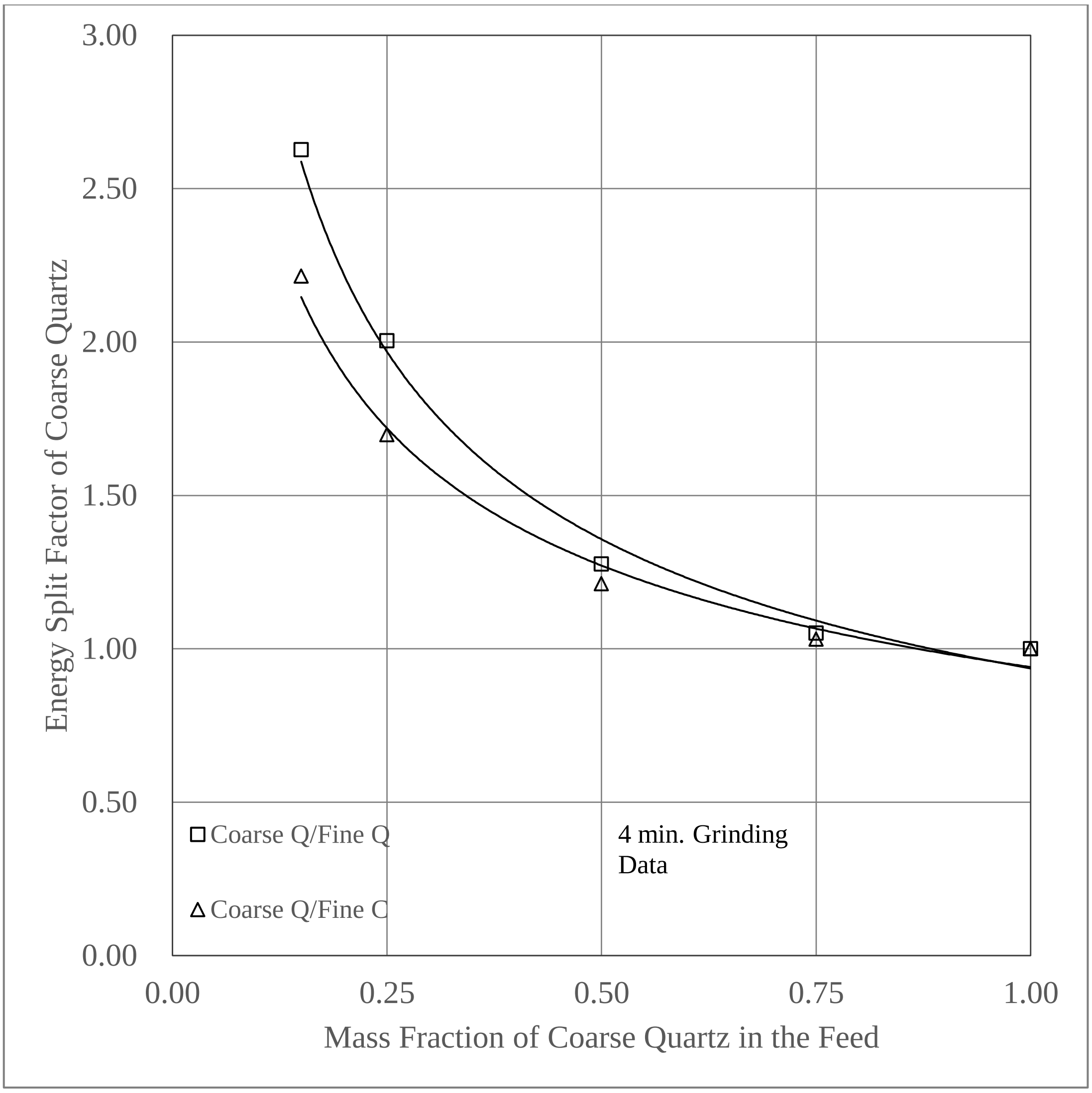


Figure 4.15 Energy split factor of coarse (-1.18+0.85 mm) quartz when ground alone and in a mixture with fine (-106 μm) quartz or calcite for four-minute dry grinding.

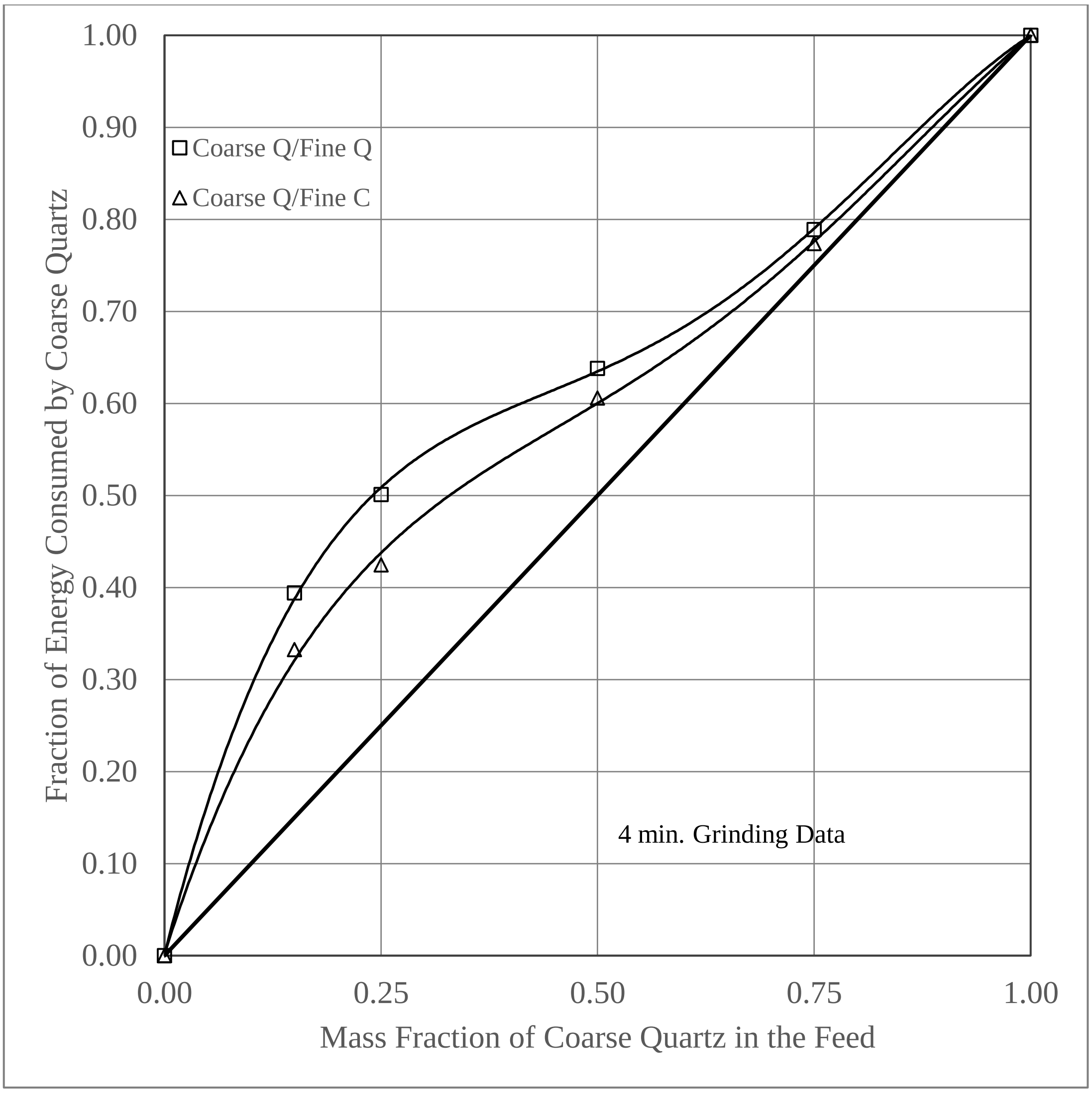


Figure 4.16 Fraction of energy consumed by coarse quartz (-1.18+0.85 mm) as a function of its mass fraction when mixed with fine quartz or calcite (-106 μm) for four- minute dry grinding.

# CHAPTER 5

# CONCLUSIONS

The following main conclusions can be drawn from the results of this investigation:

1. Batch grinding kinetic experiments showed that breakage rate of closely-sized (-1.18+0.85 mm) quartz in a mixture with fine quartz or calcite (-106 μm) was time-independent but environment-dependent.

2. The breakage of hard coarse quartz occurred more rapidly in the presence of fine materials irrespective of whether the fine particles were soft (calcite) or hard (quartz), particularly if the proportion of fines in the mixture exceeded 50% by mass. This could be explained by the probabilities of coarse and fine particles getting nipped by the grinding balls and also the energy absorbed by the particles of different kind before fracture.

3. It was observed that increase in the breakage rate of the coarse size fraction of quartz was lower if it was ground with soft fine particles as compared to the presence of hard (quartz) fine component in the mixture. This could have resulted from that a considerably greater portion of the impact energy applied by the grinding media was consumed by the soft fine material as compared to the case when hard fine quartz was used in the mixture with hard coarse quartz. Thus, it can be concluded that preferential grinding does occur in mixture grinding.

4. Similar experiments were also conducted by adding mono-size (-150+106 μm) calcite or quartz as a fine component instead of -106 μm fines. In the same way, for the same coarse-to-fine ratios, the magnitude of increase in the breakage rate of the coarse quartz was again lower when soft calcite was used as the fine

component rather than fine quartz. Additionally, if -150+106 μm of particles were used as the fine component, the variation of the breakage rate of the coarse component from its breakage rate when ground alone became smaller.

5. Wet tests gave the same trend as it was indicated by the dry grinding test results, that is, breakage of hard coarse particles occurred more rapidly in the presence of fine material, and as the fraction of the fine particles increased in the mixture the breakage rate increased as well. Additionally, the marked deviations of wet grinding results compared to the dry grinding results for all compositions tested indicated that wet grinding led to higher rates of breakage.

6. Although, the addition of the fine particles changed breakage rate function of coarse particles, the pattern of the breakage was not affected. The breakage distribution functions remained invariant for all tests, indicating that the breakage distribution function was environment-independent. Besides, it was found that the cumulative breakage distribution function of the coarse quartz remained self-similar irrespective of being in mixture with fine quartz or fine calcite, when both of the tests were conducted under identical mill operation conditions.

7. Energy split consideration results indicated that the split factor values of the coarse quartz for a given coarse-to-fine ratios were lower when the fine material in the mill feed is soft calcite rather than hard quartz. Fine calcite consumed more energy than fine quartz when ground in mixture with coarse quartz.

# REFERENCES

Acar, C. (2013). Investigation of particle breakage parameters in locked-cycle ball milling. Ph.D. Thesis, Mining Engineering Department, Middle East Technical University, Ankara, Turkey

Anderson, T. L. (2005). Fracture mechanics: fundamentals and applications. 3rd Edition, CRC Press, USA, 640 p.

Austin, L. G., Bagga, P. (1981). An analysis of fine dry grinding in ball mills. Powder Technology, 28, 83-90.

Austin, L. G., Bagga, P., Celik, M. (1981). Breakage properties of some materials in a laboratory ball mill. Powder Technology, 28, 50-243.

Austin, L. G., Klimpel, R. R., Luckie, P. T. (1984). Process engineering of size reduction: Ball milling. Society of mining engineering, AIME. 561 p.

Bilgili, E., Scarlet, B. (2005). Population balance modeling of non-linear effects in milling processing. Powder Technology, 153, 59-71.

Bond, F. C. (1952). The third theory of comminution. Transactions AIME, 193, 484-494.

Celik, M. S. (1988). Acceleration of breakage rate of anthracite during grinding in a ball mill. Powder Technology, 54, 227-233.

Charles, R. J. (1957). Energy-size reduction relationships in comminution. Transactions AIME, Mining Engineering, 9, 80-88.

Fuerstenau, D.W., Sullivan Jr., D. A. (1962). Comminution of mixtures in ball mills. Transaction, SME/AIME, 223, 152–157.

Fuerstenau, D. W., Abouzeid, A.-Z. M. (1991). Effect of fine particles on the kinetics and energetics of grinding coarse particles. International Journal of Mineral Processing, Vol. 31, 151-162.

Fuerstenau, D. W., Abouzeid, A.-Z. M. (2002). The energy efficiency of ball milling in comminution. International Journal of Mineral Processing, Vol. 67, 161-185.

Fuerstenau, D. W., Abouzeid, A.-Z. M., Phatak, P. B. (2010). Effect of particulate environment on the kinetics and energetics of dry ball milling. International Journal of Mineral Processing, Vol. 97, 52-58.

Fuerstenau, D. W., Phatak, P. B., Kapur, P. C., Abouzeid, A.-Z. M. (2011). Simulation of the grinding of coarse/fine (heterogeneous) systems in a ball mill. International Journal of Mineral Processing, Vol. 99, 32-38.

Gupta, A.; Yan, D. S. (2006). Mineral Processing Design and Operation an Introduction. Elsevier, Amsterdam, The Netherlands.

Herbst, J. A., Fuerstenau, W. D. (1973). Mathematical simulation of dry ball milling using specific power information. Trans. Society of Mining Engineers, AIME, 254, 343-348.

Herbst, J. A., Fuerstenau, W. D. (1980). Scale-up procedure for continues grinding mill design using population balance model. International Journal of Mineral Processing, 7, 1-31.

Hosten, C., Avsar C. (1998). Grindability of mixtures of cement clinker and trass. Cement and Concrete Research, Vol. 28, No. 11, 1519-1524.

Kanda, Y., Murata, H., Honma, T. (1989). Grinding of mixtures of coal and iron ore in order to prepare material for coal liquefaction. Powder Technology, 58, 175-180.

Kapur, P. C., Fuerstenau, W. D. (1988). Energy split in multicomponent grinding. International Journal of Mineral Processing, 24, 125-142.

Lynch, A. J. (1977). Mineral crushing and grinding circuits; their simulation, optimization, design and control. Elsevier, Amsterdam, The Netherlands.

Phatak, P.B. (1990). Kinetics and energetics of the dry ball milling of coarse-fine particle mixtures. M.Sc. Thesis, Department of Materials Science and Mineral Engineering, University of California, Berkeley, California, U.S.A.

Rajamani, R. K., Guo, D. (1992). Acceleration and deceleration of breakage rates in wet ball mills. International Journal of Mineral Processing, Vol. 34, 103-118.

Somasundaran, P., Fuerstenau, D.W. (1963). Preferential energy consumption in tumbling mills. Transaction, SME/AIME, 226, 132–134.

Tangsathitkulchai, C. (2002). Acceleration of particle breakage rates in wet batch ball milling. Powder Technology, 124, 67-75.

Tromans, D. (2008). Mineral comminution: Energy efficiency considerations. Minerals Engineering, 21, 613-620.

Venkataraman, K. S., Fuerstenau, D. W. (1984). Application of the population balance model to the grinding of mixtures of minerals. Powder Technology, 39, 133-142.

Verma, R., Rajamani, R. K. (1995). Environment-dependent breakage rates in ball mill. Powder Technology, 84, 127-137.

Wills, B. A., Napier-Munn T. J. (2006). Wills' mineral processing technology: An introduction to the practical aspects of ore treatment and mineral recovery. 7th edition, Elsevier Science and Technology Books, 108-146 pp.

# APPENDIX A

## PARTICLE SIZE DISTRIBUTIONS OF MINERALS AFTER SHORT-TIME BATCH GRINDING TESTS

Table A.1 Cumulative weight percent passing after batch dry grinding of coarse quartz (-1.18+0.85 mm) alone

| Size, µm | Cumulative Weight % Passing | | | | |
|---|---|---|---|---|---|
| | 0.5 min | 1 min | 2 min | 3 min | 4 min |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 10.37 | 16.55 | 26.22 | 32.16 | 37.76 |
| 600 | 5.04 | 8.30 | 13.26 | 16.63 | 19.93 |
| 425 | 2.81 | 4.56 | 7.48 | 9.31 | 11.31 |
| 300 | 1.88 | 3.14 | 5.12 | 6.44 | 7.79 |
| 212 | 1.36 | 2.27 | 3.75 | 4.80 | 5.77 |
| 150 | 1.01 | 1.69 | 2.79 | 3.61 | 4.32 |
| 106 | 0.75 | 1.28 | 2.16 | 2.77 | 3.27 |

Table A.2 Cumulative weight percent passing after batch dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and fine quartz (-106 µm) at 3:1 C:F ratio

| Size, µm | Cumulative Weight % Passing | | | | |
|---|---|---|---|---|---|
| | 0.5 min | 1 min | 2 min | 3 min | 4 min |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 8.58 | 15.05 | 24.39 | 32.00 | 39.27 |
| 600 | 4.11 | 6.92 | 11.73 | 16.19 | 20.03 |
| 425 | 2.28 | 4.15 | 6.86 | 9.60 | 11.91 |
| 300 | 1.58 | 2.79 | 4.67 | 6.50 | 8.13 |
| 212 | 1.17 | 2.06 | 3.40 | 4.78 | 5.93 |
| 150 | 0.86 | 1.54 | 2.47 | 3.51 | 4.37 |
| 106 | 0.52 | 1.02 | 1.79 | 2.52 | 3.19 |

Table A.3 Cumulative weight percent passing after batch dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and fine quartz (-106 µm) at 1:1 C:F ratio

| Size, µm | Cumulative Weight % Passing | | | | |
|---|---|---|---|---|---|
| | 0.5 min | 1 min | 2 min | 3 min | 4 min |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 11.12 | 17.68 | 27.90 | 37.25 | 45.40 |
| 600 | 5.47 | 8.38 | 14.20 | 19.56 | 24.32 |
| 425 | 3.02 | 4.87 | 7.88 | 10.88 | 14.15 |
| 300 | 2.13 | 3.42 | 5.55 | 7.52 | 9.70 |
| 212 | 1.62 | 2.54 | 4.12 | 5.57 | 7.19 |
| 150 | 1.10 | 1.80 | 2.99 | 4.09 | 5.32 |
| 106 | 0.69 | 1.16 | 1.87 | 2.74 | 3.72 |

Table A.4 Cumulative weight percent passing after batch dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and fine quartz (-106 µm) at 1:3 C:F ratio

| Size, µm | Cumulative Weight % Passing | | | | |
|---|---|---|---|---|---|
| | 0.5 min | 1 min | 2 min | 3 min | 4 min |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 17.47 | 24.29 | 39.58 | 51.14 | 61.33 |
| 600 | 8.10 | 12.46 | 22.48 | 30.59 | 38.67 |
| 425 | 4.67 | 7.42 | 14.08 | 18.83 | 23.94 |
| 300 | 3.46 | 5.34 | 10.25 | 13.74 | 17.29 |
| 212 | 2.55 | 3.99 | 8.01 | 10.55 | 13.12 |
| 150 | 1.73 | 2.66 | 5.66 | 7.50 | 9.54 |
| 106 | 1.07 | 1.83 | 3.05 | 4.45 | 6.16 |

Table A.5 Cumulative weight percent passing after batch dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and fine quartz (-106 μm) at 1:5.7 C:F ratio

| **Size, μm** | **Cumulative Weight % Passing** | | | | |
|---|---|---|---|---|---|
| | **0.5 min** | **1 min** | **2 min** | **3 min** | **4 min** |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 20.60 | 32.12 | 48.76 | 62.39 | 71.23 |
| 600 | 10.68 | 17.12 | 28.68 | 40.39 | 48.92 |
| 425 | 6.38 | 10.03 | 17.96 | 25.88 | 33.71 |
| 300 | 4.76 | 7.07 | 12.78 | 18.85 | 25.22 |
| 212 | 3.46 | 5.13 | 9.57 | 14.33 | 19.34 |
| 150 | 2.36 | 3.66 | 6.86 | 10.31 | 13.98 |
| 106 | 1.50 | 2.60 | 4.64 | 6.76 | 8.99 |

Table A.6 Cumulative weight percent passing after batch dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and fine calcite (-106 μm) at 3:1 C:F ratio

| **Size, μm** | **Cumulative Weight % Passing** | | | | |
|---|---|---|---|---|---|
| | **0.5 min** | **1 min** | **2 min** | **3 min** | **4 min** |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 9.85 | 16.26 | 24.42 | 31.80 | 38.69 |
| 600 | 4.84 | 7.70 | 11.93 | 16.47 | 20.14 |
| 425 | 2.75 | 4.24 | 6.99 | 9.17 | 11.44 |
| 300 | 1.87 | 2.85 | 4.65 | 6.29 | 7.82 |
| 212 | 1.33 | 2.03 | 3.40 | 4.57 | 5.70 |
| 150 | 0.97 | 1.51 | 2.50 | 3.32 | 4.18 |
| 106 | 0.72 | 1.12 | 1.86 | 2.42 | 3.06 |

Table A.7 Cumulative weight percent passing after batch dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and fine calcite (-106 µm) at 1:1 C:F ratio

| **Size, µm** | **Cumulative Weight % Passing** | | | | |
|---|---|---|---|---|---|
| | **0.5 min** | **1 min** | **2 min** | **3 min** | **4 min** |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 10.25 | 16.66 | 27.35 | 37.35 | 43.70 |
| 600 | 4.90 | 7.76 | 13.44 | 19.30 | 23.70 |
| 425 | 2.58 | 4.60 | 7.91 | 11.07 | 13.26 |
| 300 | 1.75 | 3.08 | 5.28 | 7.46 | 9.13 |
| 212 | 1.25 | 2.24 | 3.84 | 5.46 | 6.65 |
| 150 | 0.95 | 1.67 | 2.85 | 4.05 | 4.89 |
| 106 | 0.65 | 1.18 | 2.11 | 2.97 | 3.57 |

Table A.8 Cumulative weight percent passing after batch dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and fine calcite (-106 µm) at 1:3 C:F ratio

| **Size, µm** | **Cumulative Weight % Passing** | | | | |
|---|---|---|---|---|---|
| | **0.5 min** | **1 min** | **2 min** | **3 min** | **4 min** |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 12.23 | 20.69 | 34.22 | 44.93 | 55.30 |
| 600 | 5.71 | 10.15 | 17.74 | 24.74 | 32.79 |
| 425 | 3.01 | 5.34 | 10.20 | 14.96 | 19.24 |
| 300 | 2.00 | 3.60 | 6.91 | 10.16 | 13.39 |
| 212 | 1.43 | 2.63 | 5.08 | 7.54 | 9.84 |
| 150 | 1.07 | 1.93 | 3.80 | 5.63 | 7.28 |
| 106 | 0.80 | 1.38 | 2.64 | 3.98 | 5.27 |

Table A.9 Cumulative weight percent passing after batch dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and fine calcite (-106 µm) at 1:5.7 C:F ratio

| **Size, µm** | **Cumulative Weight % Passing** | | | | |
|---|---|---|---|---|---|
| | **0.5 min** | **1 min** | **2 min** | **3 min** | **4 min** |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 16.64 | 26.38 | 41.80 | 53.62 | 65.02 |
| 600 | 7.66 | 13.32 | 23.51 | 32.40 | 42.11 |
| 425 | 4.17 | 7.40 | 13.48 | 20.29 | 26.78 |
| 300 | 2.80 | 4.98 | 9.35 | 14.36 | 19.15 |
| 212 | 2.05 | 3.62 | 6.86 | 10.57 | 14.13 |
| 150 | 1.49 | 2.71 | 5.09 | 7.80 | 10.43 |
| 106 | 1.07 | 1.93 | 3.50 | 5.46 | 7.58 |

Table A.10 Cumulative weight percent passing after batch wet grinding of coarse quartz (-1.18+0.85 mm) alone

| **Size, µm** | **Cumulative Weight % Passing** | | | | |
|---|---|---|---|---|---|
| | **0.5 min** | **1 min** | **2 min** | **3 min** | **4 min** |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 9.58 | 17.16 | 26.05 | 34.85 | 41.29 |
| 600 | 4.69 | 8.46 | 12.96 | 18.69 | 22.56 |
| 425 | 2.62 | 4.75 | 7.37 | 11.15 | 13.78 |
| 300 | 1.80 | 3.25 | 5.16 | 8.02 | 10.03 |
| 212 | 1.34 | 2.41 | 3.86 | 6.14 | 7.75 |
| 150 | 1.06 | 1.86 | 2.97 | 4.81 | 6.10 |
| 106 | 0.86 | 1.47 | 2.35 | 3.83 | 4.85 |

Table A.11 Cumulative weight percent passing after batch wet grinding of a mixture of coarse quartz (-1.18+0.85 mm) and fine calcite (-106 μm) at 1:1 C:F ratio

| Size, μm | Cumulative Weight % Passing | | | | |
|---|---|---|---|---|---|
| | **0.5 min** | **1 min** | **2 min** | **3 min** | **4 min** |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 11.79 | 19.32 | 32.71 | 40.25 | 49.16 |
| 600 | 5.44 | 9.24 | 16.57 | 21.10 | 27.70 |
| 425 | 2.84 | 5.20 | 9.43 | 12.91 | 16.16 |
| 300 | 1.89 | 3.41 | 6.33 | 8.73 | 11.14 |
| 212 | 1.35 | 2.49 | 4.60 | 6.41 | 8.17 |
| 150 | 1.02 | 1.85 | 3.44 | 4.74 | 6.02 |
| 106 | 0.73 | 1.38 | 2.52 | 3.50 | 4.40 |

Table A.12 Cumulative weight percent after batch wet grinding of a mixture of coarse quartz (-1.18+0.85 mm) and fine calcite (-106 μm) at 1:5.7 C:F ratio

| Size, μm | Cumulative Weight % Passing | | | | |
|---|---|---|---|---|---|
| | **0.5 min** | **1 min** | **2 min** | **3 min** | **4 min** |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 20.04 | 31.15 | 50.19 | 61.81 | 72.08 |
| 600 | 9.71 | 16.55 | 29.10 | 38.82 | 48.40 |
| 425 | 5.43 | 9.75 | 17.39 | 25.92 | 33.20 |
| 300 | 3.74 | 6.68 | 12.16 | 18.48 | 24.18 |
| 212 | 2.81 | 4.95 | 8.92 | 13.90 | 18.25 |
| 150 | 2.22 | 3.71 | 6.67 | 10.34 | 13.52 |
| 106 | 1.14 | 2.42 | 4.60 | 6.96 | 9.33 |

Table A.13 Cumulative weight percent passing after batch dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and narrowly size fine quartz (-150+106 µm) at 1:3 C:F ratio

| **Size, µm** | **Cumulative Weight % Passing** | | | | |
|---|---|---|---|---|---|
| | **0.5 min** | **1 min** | **2 min** | **3 min** | **4 min** |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 12.21 | 18.33 | 29.19 | 37.35 | 43.77 |
| 600 | 4.83 | 7.86 | 14.23 | 19.43 | 23.87 |
| 425 | 2.79 | 4.30 | 7.86 | 11.20 | 14.30 |
| 300 | 2.01 | 2.94 | 5.36 | 7.67 | 9.90 |
| 212 | 1.60 | 2.15 | 3.91 | 5.59 | 7.26 |
| 150 | 0.87 | 1.55 | 2.92 | 4.23 | 5.43 |

Table A.14 Cumulative weight percent passing after batch dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and narrowly size fine quartz (-150+106 µm) at 1:5.7 C:F ratio

| **Size, µm** | **Cumulative Weight % Passing** | | | | |
|---|---|---|---|---|---|
| | **0.5 min** | **1 min** | **2 min** | **3 min** | **4 min** |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 12.80 | 20.87 | 32.65 | 41.26 | 48.84 |
| 600 | 4.70 | 9.23 | 16.63 | 21.46 | 27.52 |
| 425 | 2.65 | 5.18 | 10.14 | 12.73 | 16.87 |
| 300 | 1.76 | 3.62 | 7.50 | 8.92 | 11.96 |
| 212 | 1.28 | 2.69 | 5.86 | 6.62 | 9.00 |
| 150 | 0.98 | 1.82 | 3.27 | 4.67 | 6.21 |

Table A.15 Cumulative weight percent passing after batch dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and narrowly size fine calcite (-150+106 μm) at 1:3 C:F ratio

| Size, μm | Cumulative Weight % Passing | | | | |
|---|---|---|---|---|---|
| | 0.5 min | 1 min | 2 min | 3 min | 4 min |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 10.65 | 17.13 | 26.68 | 33.71 | 40.70 |
| 600 | 4.01 | 7.23 | 12.69 | 16.86 | 21.34 |
| 425 | 2.20 | 3.98 | 7.19 | 9.56 | 12.38 |
| 300 | 1.50 | 2.77 | 5.06 | 6.54 | 8.55 |
| 212 | 1.10 | 2.06 | 3.84 | 4.80 | 6.28 |
| 150 | 0.79 | 1.40 | 2.49 | 3.56 | 4.65 |

Table A.16 Cumulative weight percent after passing batch dry grinding of a mixture of coarse quartz (-1.18+0.85 mm) and narrowly size fine calcite (-150+106 μm) at 1:5.7 C:F ratio

| Size, μm | Cumulative Weight % Passing | | | | |
|---|---|---|---|---|---|
| | 0.5 min | 1 min | 2 min | 3 min | 4 min |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 11.84 | 17.07 | 30.92 | 38.97 | 45.23 |
| 600 | 4.28 | 7.50 | 15.05 | 19.59 | 24.18 |
| 425 | 2.31 | 4.18 | 9.09 | 11.41 | 14.20 |
| 300 | 1.53 | 2.89 | 6.72 | 7.96 | 9.88 |
| 212 | 1.15 | 2.13 | 5.40 | 5.91 | 7.28 |
| 150 | 0.83 | 1.48 | 2.82 | 4.17 | 5.34 |

Table A.17 Cumulative weight percent passing after batch dry grinding of coarse calcite (-1.18+0.85 mm) alone

| Size, µm | Cumulative Weight % Passing | | | | |
|---|---|---|---|---|---|
| | 0.5 min | 1 min | 2 min | 3 min | 4 min |
| 1180 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 850 | 19.38 | 29.99 | 51.41 | 60.49 | 72.46 |
| 600 | 10.88 | 16.38 | 32.42 | 40.70 | 51.78 |
| 425 | 6.95 | 10.48 | 20.61 | 27.00 | 35.77 |
| 300 | 4.86 | 7.16 | 14.78 | 20.09 | 26.82 |
| 212 | 3.59 | 5.33 | 11.00 | 15.29 | 20.56 |
| 150 | 2.70 | 3.99 | 8.24 | 11.62 | 15.75 |
| 106 | 2.06 | 3.02 | 6.17 | 8.79 | 11.98 |

Table A.18 Cumulative weight percent passing after batch dry grinding of fine quartz (-150+106 µm) alone

| Size, µm | Cumulative Weight % Passing | | | | |
|---|---|---|---|---|---|
| | 0.5 min | 1 min | 2 min | 3 min | 4 min |
| 150 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 106 | 8.27 | 13.54 | 19.31 | 24.04 | 27.13 |

Table A.19 Cumulative weight percent passing after batch dry grinding of fine calcite (-150+106 µm) alone

| Size, µm | Cumulative Weight % Passing | | | | |
|---|---|---|---|---|---|
| | 0.5 min | 1 min | 2 min | 3 min | 4 min |
| 150 | 100.00 | 100.00 | 100.00 | 100.00 | 100.00 |
| 106 | 11.04 | 15.92 | 21.16 | 29.50 | 32.65 |

## APPENDIX B

## SIZE DISTRIBUTIONS OF FINE COMPONENTS

Table B.1 Size distribution of fine quartz (-106 µm)

| Size, µm | Weight (g) | Weight (%) | Cum. Weight % Passing |
|---|---|---|---|
| 106 | 0.00 | 0.00 | 100.00 |
| 75 | 4.31 | 10.43 | 89.57 |
| 53 | 8.59 | 20.78 | 68.79 |
| 38 | 5.58 | 13.50 | 55.29 |
| -38 | 22.85 | 55.29 | 0.00 |
| Total | 41.33 | | |

Table B.2 Size distribution of fine calcite (-106 µm)

| Size, µm | Weight (g) | Weight (%) | Cum. Weight % Passing |
|---|---|---|---|
| 106 | 0.00 | 0.00 | 100.00 |
| 75 | 16.39 | 41.22 | 58.78 |
| 53 | 5.83 | 14.66 | 44.11 |
| 38 | 3.30 | 8.30 | 35.81 |
| -38 | 14.24 | 35.81 | 0.00 |
| Total | 39.76 | | |

74

## APPENDIX C

## ENERGY SPLIT FACTORS AND FRACTION OF ENERGY CONSUMPTION

Table C.1 Energy split factors and fraction of energy consumed by coarse (-1.18+0.85 mm) quartz when mixed with fine (-106 μm) quartz or calcite

| 4 min Grinding Data | | | | |
|---|---|---|---|---|
| **Material Mixture** | **C:F Ratio** | **Mass Fraction of Coarse** | **Energy Split Factor of Coarse Quartz** | **Fraction of Energy Consumed by Coarse Quartz** |
| **Coarse Quartz Fine Quartz** | 1:0 | 1.00 | 0.00 | 1.00 |
| | 3:1 | 0.75 | 1.05 | 0.79 |
| | 1:1 | 0.50 | 1.28 | 0.64 |
| | 1:3 | 0.25 | 2.00 | 0.50 |
| | 1:5.7 | 0.15 | 2.63 | 0.39 |
| **Coarse Quartz Fine Calcite** | 1:0 | 1.00 | 1.03 | 1.00 |
| | 3:1 | 0.75 | 1.03 | 0.77 |
| | 1:1 | 0.50 | 1.21 | 0.61 |
| | 1:3 | 0.25 | 1.70 | 0.42 |
| | 1:5.7 | 0.15 | 2.22 | 0.33 |