# INVESTIGATION OF PARAMETERS AFFECTING THE DRYING RATE OF SANITARY WARES

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# ABSTRACT

## INVESTIGATION OF PARAMETERS AFFECTING THE DRYING RATE OF SANITARY WARES

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The influence of drying parameters namely residence time before drying, drying temperature, drying time, relative humidity, and slip recipe on the drying rate of slip cast sanitary wares, predominantly lavatories and toilet closets, was studied.

The drying temperatures were changed from 80 $^{o}$C to 110 $^{o}$C with an increment of 10 $^{o}$C. The drying time was changed from 10 to 7 h with a decrease of 1 hour. Relative humidity of the environment was changed from 60 to 75 %. The percent weight loss, percent residual moisture and the percent shrinkage of the samples were determined by weighing and measuring the samples before and after the tests. The percent weight loss was within the range of 6.5 to 6.6 % after holding the as cast samples for 6 hours at ambient casting shop conditions while it was within the range of 17.96 to 18.10 % when subsequently dried for 10 hours at 110 $^{o}$C in the dryer. The percent shrinkage was within the range of 2.9 - 3.0 % after holding the as cast samples 6 h at ambient laboratory conditions. No shrinkage

was observed in the sample when it was subsequently dried for 10 hours at 110 $^{o}$C in the dryer.

Optimum moisture content of dried wares was obtained after drying for 8 hours at 100 $^{o}$C in the dryer. It has been seen that the relative humidity of the dryer at the beginning of the drying should be lower than 75 %. As the non-plastic content in the recipe of the sanitary ware slip increased, drying shrinkage and residual moisture content decreased.

The results of this study showed that through increasing the residence time up to 6h with a casting shop environment of approximately 30 $^{o}$C and 60 % relative humidity, the drying time could be safely reduced from 10 h to 8 h with a drying temperature of 100 $^{o}$C for the test plates.

The same approach can be used for more complex shapes, e.g., WC closets, basins, tanks etc. in Eczacıbaşı Vitra plant. Once the drying time was reduced, the amount of natural gas per ware would be reduced to a certain extent. Aside from that the reduction in the drying time would increase the quantity of the drying cycles per week so that more wares could be dried.

When all these observations were taken into account, this thesis study could also be utilized by other sanitary ware producing companies whose processes require slip cast drying.

Keywords: drying rate, drying shrinkage, moisture, sanitary ware, slip casting.

# ÖZ

## SERAMİK SAĞLIK GEREÇLERİNDE KURUTMA HIZINI ETKİLEYEN PARAMETRELERİN İNCELENMESİ

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Bu çalışmada; kurutma fırını öncesi bekleme süresi, kurutma sıcaklığı, kurutma süresi, bağıl nem ve çamur reçetesi gibi parametrelerin özellikle lavabo ve klozet gibi sağlık gereçleri ürünlerinin kurutma hızına olan etkisi incelenmiştir.

Kurutma sıcaklığı 10 $^{o}$C' lik artışla 80-110 $^{o}$C arasında değiştirilmiştir. Kurutma süresi 7-9 saat aralığında 1 saatlik artışla değiştirilmiştir. Kurutma başlangıcındaki bağıl nem oranı %60'tan %75'e yükseltilmiştir. Yüzde ağırlık kaybı, yüzde nem miktarı ve yüzde kuru küçülme değerleri, yapılan testlerin öncesinde ve sonrasında numunelerin ağırlıkları ve boyutları ölçülerek belirlenmiştir. Numuneler dökümhane şartlarında 6 saat kurutulduğunda ağırlık kaybı %6,5-6,6 arasında değişirken, endüstriyel kurutma fırınlarında 110 $^{o}$C'de 10 saat kurutulduğunda % 17,96-18,10 arasında değişmektedir. Yüzde kuru küçülme, dökümhane şartlarında 6 saat kurutma uygulandığında % 2,9-3,0 arasında değerler

almaktadır. 110 $^{o}$C'de 10 saat kurutma uygulandığında kuru küçülme değerlerinde belirgin bir farklılık oluşmamıştır.

En uygun bağıl nem miktarı 100 $^{o}$C'de 8 saat kurutma uygulanarak belirlenmiştir. Bağıl nemin kurutma başlangıcında % 75'ten daha fazla olmaması gerektiğine karar verilmiştir. Çamur reçetesinde plastik olmayan hammadde miktarı arttırıldığında, kuruma küçülmesi ve nem miktarında azalma görülmüştür.

Sonuç olarak; sıcaklığı ve bağıl nemi ortalama 30 $^{o}$C ve 60 % olan dökümhane şartlarında, deney plakaları için kurutma öncesi bekleme süresi 6 saate kadar çıkartıldığında, 100 $^{o}$C' lik bir kurutma fırınında kurutma sıcaklığı 10 saatten 8 saate kadar güvenli bir şekilde düşürülebilir.

Aynı yaklaşım Eczacıbaşı Yapı Gereçleri Vitra tesislerinde üretilen klozet, lavabo ve rezervuar gibi daha kompleks şekilli ürünlerde de kullanılabilir. Kurutma süresi düşürüldüğünde, ürün başına harcanan doğal gaz miktarı belirli bir değere kadar azaltılabilir. Bunun yanı sıra, kurutma süresindeki azalma haftalık kurutma şarj sayısını arttıracağından, daha fazla sayıda ürünün kurutulması sağlanacaktır.

Tüm bu çalışmalar dikkate alındığında, bu tez çalışması seramik sağlık gereçleri üretimi yapan diğer firmalara da ışık tutacaktır.

Anahtar Kelimeler: kuruma hızı, kuruma küçülmesi, nem, sağlık gereçleri, çamur dökümü.

To My Wife

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# TABLE OF CONTENTS

Page Number

# LIST OF TABLES

Tables

# LIST OF FIGURES

Figures

# LIST OF SYMBOLS

Symbols

| | |
|---|---|
| $b$ | the barometric pressure of the surrounding atmosphere |
| $B.D$ | bulk density |
| $c$ | the coefficient determining the evaporation as a function of the circulation rate of the gases on the surface |
| $C_d$ | water concentration at the dry phase of the body |
| $C_w$ | water concentration at the wet phase of the body |
| $dM / dt$ | the rate of moisture movement |
| $F$ | the evaporation surface |
| $k$ | constant |
| $l$ | path length |
| $L_1$ | initial length of the experimental slip plates |
| $L_2$ | final length of the experimental slip plates |
| $m_{water}$ | amount of water to be added to the slip mixture |
| $p$ | permeability of the body |
| $P_p$ | the partial water vapor pressure in the surrounding gas medium |
| $P_s$ | the saturated water vapor pressure at the given temperature of the evaporation surface |
| $R_1$ | the recipe used on the production of 45 % non-plastic raw materials |
| $R_2$ | the recipe used on the production of 55 % non-plastic raw materials |
| $R_p$ | the recipe used on the production of lavatories and toilet closets |
| $R.H$ | the relative humidity of air |
| $S.G$ | specific gravity |
| $W$ | the amount of evaporating water |
| $W_1$ | weight of the plates just after casting |
| $W_2$ | weight of the plates before putting the samples into the dryer |
| $W_3$ | weight of the plates after drying |

| | |
|---|---|
| $W_i$ | weight of the plates prepared by the standard recipe just after casting |
| $W_f$ | weight of the plates prepared by the standard recipe after drying to 0 % moisture level |
| $z$ | time |
| $\eta$ | viscosity of water |

# CHAPTER 1

# INTRODUCTION

Drying is the removal of liquid from a porous material by means of its transport and evaporation into a surrounding unsaturated gas or in some cases a desiccating liquid [1]. Drying is a crucial production step in ceramic manufacturing due to the elimination of liquid from the body. When the liquid flows through a porous body in response to a gradient in pressure; at the same time, the pressure causes deformation of the solid network, and dilatation of the pores through which the liquid moves [2]. Drying must be carefully controlled, because stresses produced by differential shrinkage or gas pressure may cause defects in the product [1]. Defects such as cracks, black coring and bloating are often the cause of drying. Long drying schedules have been implemented to ensure that these defects do not occur [3]. On the other hand, a shorter drying cycle could allow an increase in the production rate of quality parts. Hence, safely changing the drying schedule requires a true understanding of the drying process.

In a drying system, heat energy must be brought to the surface of the product and vapors must be carried away. The mechanisms of evaporation and mass and thermal transport must be considered before deciding which mode of the drying process is to be considered and selected [1].

While the concept is simple, the actual process is complex, involving several different steps and possessing numerous potential problems [4]. The evaporation of processing liquids is relatively energy intensive, and drying efficiency is

always an important consideration. The drying of a ceramic material is a highly variable process, which requires accurate industrial controls of critical parameters such as relative humidity, drying speed and air temperature [5]. The evaporation speed rises with the increase in [6];

- The difference between its vapor tension in the piece and the vapor pressure in the drying air, therefore with its temperature;
- The drying air speed;
- The surface/volume ratio, or the specific surface of the product

Slow heating rates or raising the humidity around the ware prior to heating to prevent moisture loss has been commonly employed to overcome the drying problems. Once the body has achieved a uniform temperature, the humidity is lowered and drying begins. In either case, drying can be time consuming and expensive. Attempts have been made to develop new techniques that will dry the products in short time without causing any defects.

The use of microwave energy for drying has been considered as an alternative technique [7]. Conduction and convection drying are commonly used for drying ceramic product. Floors or shelves supporting the product may be heated by waste heat or steam. Convection drying is used to heat the product and remove vapors, and the air circulation may be designed to facilitate the drying of a particular size, setting, or shape. Infrared drying can be used to reduce the drying time and for the drying of coatings such as decorations and glazes. Vacuum–assisted drying reduces the partial pressure of vapors [8].

Slevin [5] studied the problems of the drying process for sanitary ware. By using temperature in the range of 40 and 120 $^{o}$C, the results obtained were similar with Bigot curves. However, shrinkage speeds were different depending on the thermal cycle [5]. Clausen and Fish [9] monitored the percent moisture content until the

critical shrinkage point of the wares. It could exonerate the drying process from becoming a potential source of defects [9].

Although similar studies have been reported in the literature on the factors affecting the drying rate, the data are sparse and could not be applied to all slip cast sanitary wares. Therefore it is necessary to conduct a study to understand the effects of temperature, time, relative humidity and composition of the slip on drying rate. Any information in this manner would be an asset to ceramic technology and literature. Therefore studies conducted on the drying process have technological and scientific significance.

The purpose of this study was to determine the parameters affecting the drying rate of a sanitary ware produced by slip casting. Effects of temperature, time, relative humidity and composition of slip on drying rate were investigated. The aim was to decrease the drying time and drying temperature of the as cast slip products without causing any drying defects and to minimize the natural gas consumption in drying process. Results of this thesis work were compared and discussed with the results of the studies reported in the literature for similar material systems.

# CHAPTER 2

# LITERATURE REVIEW

## 2.1 THE DRYING PROCESS

When the liquid flows through a porous body in response to a gradient in pressure; the pressure causes deformation of the solid network, and dilatation of the pores through which the liquid moves [2]. If the moisture is transferred unevenly from the surface and the deeper layers of the part, there is uneven shrinkage and stresses occur in the mixture, leading to deformation and cracking. Uniform moisture transfer and shrinkage in the surface and deeper layers of the mixture can only be ensured if as much moisture flows along the capillaries as evaporates from the surface [10].

Drying process is complex taking place in two main stages. Firstly the constant rate period when the water loss is from the surface and shrinkage occurs equal in volume to that of the water lost. Secondly the falling rate period when water evaporates inside the body and little or no shrinkage occurs [11].

### 2.1.1 STAGES OF DRYING

The removal of water from ceramic ware can take place only at the surface by evaporation. The water on the interior of the article must travel to the surface by seeping through interconnected pores. Heating accelerates both process – evaporation and seepage toward the surface. Since the individual particles that make up the wet formed ware are held apart by thin layers of water the removal of

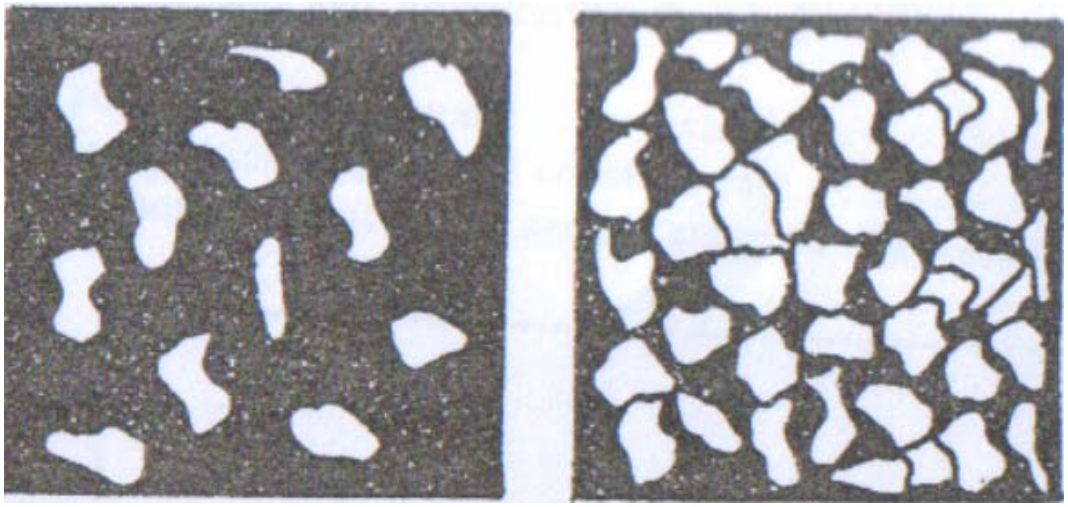
this water will cause the particles to pull together, resulting in an overall decrease in the volume of the piece [12].

Drying is often regarded as occurring in three stages corresponding to the ranges of liquid content for which the drying rate is increasing, constant and decreasing as discussed in detail below [8].

#### 2.1.1.1. FIRST STAGE OF DRYING

This is the first and most critical stage of drying, where water is actually removed from the ceramic body and high shrinkage is observed. A typical clay body contains about 15 – 20 % mechanical water. Prior to drying, individual grains are separated from each other by a thin film of water. As water leaves the body, the grains move closer together and eventually contact each other. This collapsing of the internal structure results in the body shrinkage [3]. The first stage of drying is called the constant rate period, because the rate of evaporation per unit area of the drying surface is independent of time [2].

The physical state of fine-grained-material when a sufficiently large amount of water is present maybe imagined to be as shown in Figure 2.1.a. Each particle is separated from its immediate neighbours by a water film of a relatively appreciable thickness. In this state, the clay or ceramic body can be easily moulded because the free water surrounding all particles acts as a cushion and permits the mass to flow, either unaided or under slight pressure [13].



( a ) ( b )

**Figure 2.1.** First stage in drying [13];

(a) The initial state with excess free water

(b) Particles just touching each other – the critical point

During the first stage of drying, the pores are full of liquid and evaporation occurs at the exterior surface of the body. The rate of evaporation is controlled by diffusion through a humid boundary layer at the surface; this layer can be millimeters thick in stagnant air, or microns thick in a fast draft. In any case the thickness of the boundary layer is generally much greater than the pore size, so lateral diffusion within the layer homogenizes the humidity near the drying surface. Since the evaporation rate depends on the difference between the average vapor pressure at the surface ($p_s$) and that at the top of the boundary layer ($p_b$), the rate of evaporation remains constant even when dry patches appear on the surface [14].

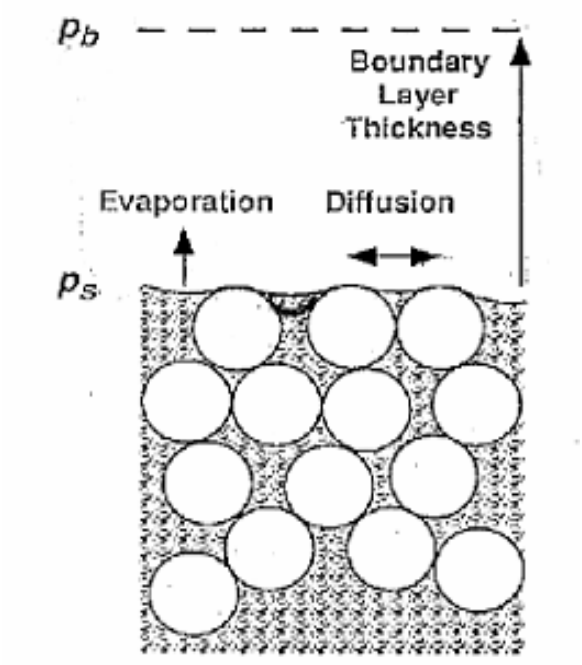



**Figure 2.2.** Rate of evaporation is controlled by diffusion through boundary layer whose thickness is much greater than the pore size [14]

During constant drying phase, the water migration speed to the surface is greater than the evaporation speed. A thin layer of water thus forms on the entire surface of the piece. In these conditions the piece brings itself to the humid air temperature [6]. All the pores remain full of liquid, and the change in volume of the body is exactly equal to the volume of liquid that evaporates. As the body contracts it becomes increasingly rigid, so more force is required make it contract; therefore the menisci deepen and pressure rises [14].

As water is lost by surface evaporation, a drawing together of the particles preserves the continuum in the mass and an over-all shrinkage occurs. The clay mass will lose water as though there were a free, surrounding water surface until the solid particles touch each other and the water within the mass can no longer maintain a continuous film as seen in Figure 2.1.b [13].

Once about half of the water has been removed and the ceramic particles are contacting each other, shrinkage has essentially ceased. At this point, drying progresses to stage two – removing the pore water [3].

#### 2.1.1.2. SECOND STAGE OF DRYING

At the critical point of drying, also known as the leather hard point, the body stops shrinking and the liquid / vapor interface invades the pores [14]. Even though the grains are contacting each other, significant amounts of water remain between the grains and inside the pores. The pore water must be removed through this tight, tortuous structure before internal temperatures reach 100 $^{o}$C, when the water can turn to steam and catastrophically crack the body [3].

As the particles become closer as given in Figure 2.3, there is a considerable reduction in the number of openings available for the passage of water and therefore the surface is no longer uniformly covered in liquid; the evaporation phenomenon begins inside the thickness of the ceramic mass with the consequent migration of vapour to the surface. The more the evaporation area moves towards the inside the greater is the reduction in the drying speed [6].

Most of the evaporation is still occurring at the exterior surface so the surface remains below the ambient temperature, and the rate of evaporation is sensitive to the ambient temperature and vapor pressure. The liquid in the pores near the surface remains in the funicular condition, so there are contiguous pathways along which evaporation can occur. At the same time, some liquid evaporates within the unsaturated pores and the vapor is transported by diffusion. Analysis of the situation involves coupled equations for flow of heat and liquid and diffusion of vapor, with transport coefficients that are generally dependent on temperature and concentration [2].

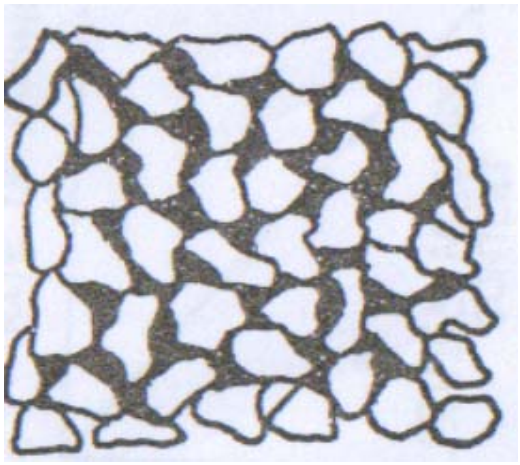


**Figure 2.3.** Second stage in drying [13]

During the second stage the body continues to lose water at a slower rate through surface evaporation but with very little additional shrinkage. The body surface is dried during stage two drying. Once the temperature reaches the water boiling point, almost all of the pore water should be removed, and stage two progresses to the final drying stage – removing residual water [3].

### 2.1.1.3. FINAL STAGE OF DRYING

This final stage in the drying is known as the falling rate period and in many bodies the change – over in drying mechanism is so well defined and the critical point can be accurately established [13]. At this stage, drying is said to enter the second falling rate period, where evaporation occurs inside the body. The temperature of the surface approaches the ambient temperature and the rate of evaporation becomes less sensitive to external conditions (temperature, relative humidity, draft rate etc.) [2].

In the final drying stage, the temperature must be increased above the boiling point of water to remove the final small amount of residual mechanical water [3].

It is important not to provoke sudden rises of the internal water vapour pressure because if this exceeds the mechanical resistance of the material the piece could break violently [6].

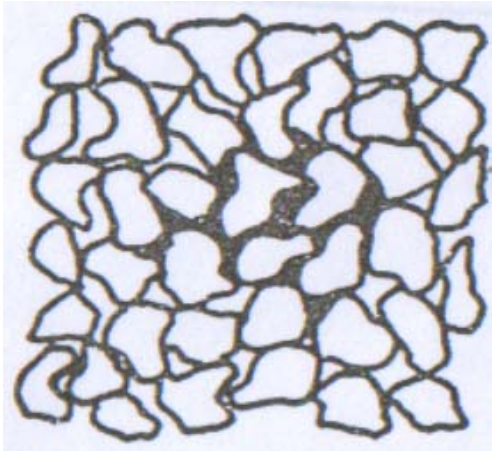


**Figure 2.4.** Final stage in drying [13]

There is no shrinkage and the ware experiences negligible weight loss during this stage. At the end of stage three, all the mechanical water is gone. The only water remaining is the chemically attached water as seen in Figure 2.4, which will be liberated during the firing process at higher temperatures [3].

### 2.1.2. STAGES OF SHRINKAGE

Drying shrinkage behavior, as given by Bigot Curves (Figure 2.5), is normally used as routine control in traditional ceramic production for testing the sensibility of clays and pastes to drying. This behavior, as a matter of fact, permits an accurate and complete characterization of slip cast bodies [15].

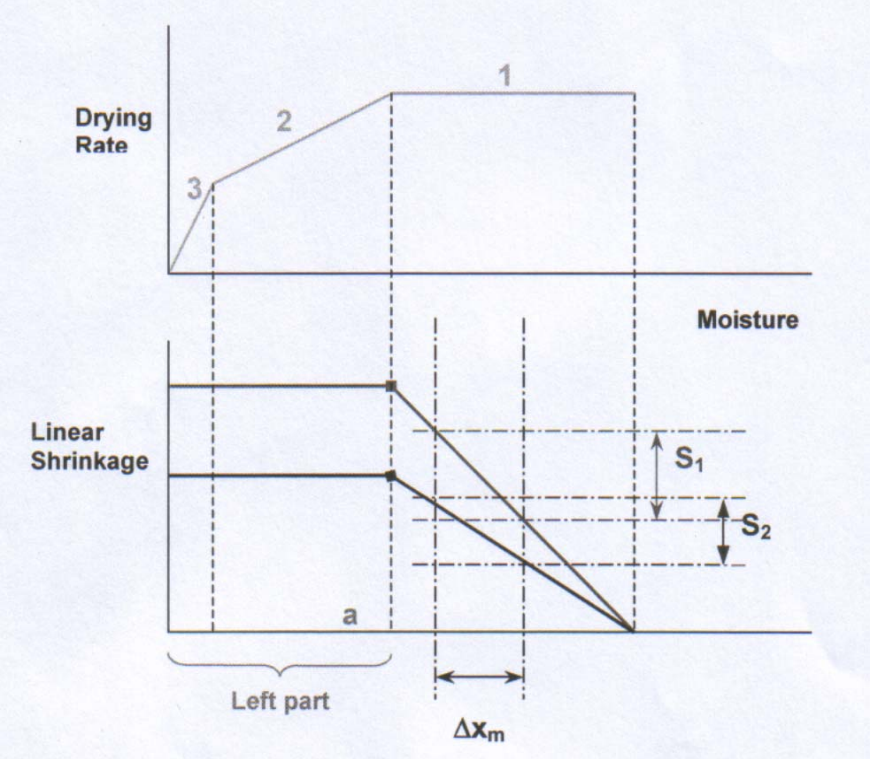



**Figure 2.5.** Bigot Curve [16]

The amount of linear drying shrinkage for $S_1$ for a defined dehydration ($\Delta X_m$) is high. So the sensitivity to drying of this mass ($S_1$) is great. In the area to the left of point a, the drying process can be increased without the green products' geometric lengths being further reduced [16].

Dimensional changes suffered by the porous structure during drying are due to stresses derived from temperature and moisture gradients existing within the body and from the capillary forces. The first ones produce differential volume changes with heating, due to differential thermal expansion of the components inside the body and moisture removal. The capillary suction forces are connected with the wetting properties of the solid by the liquid, and with the porous structure of the wet body. If shrinkage took place uniformly, as can be approximately considered

in relative dense slip cast bodies, the strain behavior will be quite simplified since the shrinkage is related only to the capillary mechanism [15].

*In the first stage of shrinkage;* the water eliminated is interstitial that flows with relative ease to the surface of the piece causing it to shrink [6]. Actually, all the shrinkage takes place during the first drying phase. During the first stage of shrinkage, the volume reduction of the green product is equal to the volume of the water emitted. When the clay particles touch each other, first shrinkage stage is completed [16].

*In the second stage of shrinkage;* shrinkage continues until the particles come into contact with each other and at this point the contraction of the piece ceases and porosity formation begins [6]. The last remaining gap water and about all the cladding water is removed in this stage. The removed cladding water has a small influence in this phase. At the end of this stage, the geometric arrangement of the particles has been completed and mass losts its plasticity [16].

*In the third shrinkage stage;* there is no shrinkage in this stage; the last remaining cladding water is removed. The volume of water given is equal to the volume increase of the pores filled with air [16].

As the meniscus recedes into the body, the exterior does not become completely dry right away, because liquid continues to flow to the outside; as long as the flux of liquid is comparable to the evaporation rate, the funicular condition is preserved. However, as the distance from the exterior to the drying front increases, the capillary pressure gradient decreases and therefore so does the flux. Eventually it becomes so slow that the liquid near the outside of the body is isolated in packets, so flow to the surface stops and liquid is removed from the body only by diffusion of its vapor [2].

## 2.2. DRYING IN SANITARYWARE INDUSTRY [6]

In the sanitaryware industry, production methods tend to follow lines that have been well established for many years. Before considering the problems that might arise in any attempt to apply flow production methods to the manufacture of sanitaryware, it was obviously very necessary to obtain as much detailed information as possible on the present practices.

Drying in the sanitaryware industry is almost always carried out in two phases:

*a) Green drying* (also called "leather-like effect"), the pieces extracted from the mould are subjected to an initial drying that makes them more consistent and easy to handle for finishing

*b) White drying*, the semi-processed products are dried to a humidity residue value of around 1%

In some industrial production cycles, especially in the more modern and automated ones that use pressure casting, drying is carried out in one single operation.

The finishing operation of pieces may be carried out immediately after demoulding, after green or white drying, but in the majority of cases it is carried out on the damp piece after green drying and before white drying.

### 2.2.1. GREEN DRYING [6]

This partial drying is done in almost all companies of the sector after extraction from the mould: the pieces cast with traditional plaster moulds, have a poor consistency and therefore cannot be handled and finished immediately. It is necessary to eliminate a part of their water content with an initial drying operation known as green drying.

During this treatment, phenomena connected with the first and, partially or totally, with the second phase of drying take place. There is a 14 – 16 % to 9 – 10 % reduction in the water content of the pieces in the case of vitreous china and a 10 - 11 % to 5 – 6 % reduction for fire clay. These humidity variations cause 2 – 3 % shrinkages for vitreous bodies and 0.5 - 1 % shrinkages for fire clay bodies. The almost total unfired shrinkage occurs during green drying so it is therefore necessary to take great care in green drying to avoid risks of breakage.

Often, in anomalous conditions and especially with articles such as in WCs, it is necessary to cover certain areas of the surface in order to reduce the drying speed and to make it uniform with the parts of the same piece characterized by slower drying rates. In this way, varying shrinkage rates and related tension that may lead to the breakage of the ceramic mass are avoided.

Green drying involves leaving the pieces extracted from the moulds on the demoulding trays in the casting area that is then heated in the next 10 - 16 hours.
The thermo-hygrometric conditions that are most commonly found during this phase are:

- temperature 33 - 36 °C
- relative humidity 40 – 60 %.

**Table 2.1.** Operated parameters of green drying at room temperature [6]

| **Plant** | **1V** | **2V** | **3V** | **4V** | **1G** |
|---|---|---|---|---|---|
| Average temperature in C | 37 | 30 | 35 | 35 | 35 |
| Average relative humidity % | 52.5 | 58.5 | 43.5 | 54 | 60 |

Table 2.1 shows the average temperature and average relative humidity recorded during green drying at room temperature in different plants: those marked V are vitreous producing plants; only the one marked G is a plant that manufactures fine fire clay sanitary ware.

### 2.2.2. WHITE DRYING [6]

White drying helps to reduce the water content of the cast piece to values at or equal to 1 %, that in the beginning is;

a) 8 – 10 % in weight for vitreous and 4 – 5 % for fire clay in the case that the sanitaryware is subjected to green drying

b) 15 – 18 % in weight for vitreous and 11 – 13 % for fire clay when drying is carried out in one single operation

All traditional plants that operate according to a manual or semi-mechanized production cycle adopt solution “a”, whilst solution “b” is almost always used in highly automated companies that employ pressure casting, making it possible to handle the damp pieces without causing deformation, right up to drying.

It is obvious that, whilst in the first case the physical phenomena tied to drying are those of the third phase, solution “b” involves the development of all three drying phases of a sanitaryware body.

## 2.3 TYPES OF INDUSTRIAL DRYERS

Heat can be supplied to the ware by convection, conduction and radiation. Convection is the usual method; especially as air movement is necessary to carry the water vapor away. Convection is induced by placing steam pipes in the floors and walls of driers, or by blowing in hot air or kiln waste gases. Ware is heated by conduction by placing it directly on a heated floor in driers but as it is a thermal insulator uniform heating cannot occur [11].

*In hot air driers*, air is used as a heating fluid and as a means to evacuate the evaporated water. The hot air may be recovered (from the kiln) or may be generated by a relative burner. Fans move and convey the air into suitable distribution canals. The optimisation of airflow, in terms of speed and distribution, is actually what discriminates between modern rapid dryers and traditional ones [6].

The fundamental operating parameters of these traditional dryers are;

- Maximum temperature of 120 °C
- Drying cycles of 20 to 24 hours in relation to the shape and thickness of the piece, the type of body and its initial humidity (green dried or not).

*In microwave drying system*, microwaves are generally reflected by electrical conductor, transmitted by electrical insulators, and absorbed by dielectrics. Liquid water behaves like a dielectric when subjected to a microwave field. When drying ceramics, the microwaves are preferentially adsorbed by the water, and the product temperature during drying may never exceed 50 $^{o}$C, that is, the high surface temperatures in conventional drying are avoided. Potential uses for microwave drying are in the processing of temperature – sensitive products, more rapid drying, drying products of large cross sections and drying products containing colloidal materials such as gels [1].

The diffusional flow rate for a given moisture gradient will be much higher near the center of a body than near the surface leading to a strong leveling process. Thus microwave-dried bodies have a completely different moisture distribution pattern with time than conventionally dried ware [7].

*The type of vacuum dryers* has a discontinuous operation and if introduced into a continuous production line they need a small inlet and outlet chamber. The dryer has been designed for drying in two phases [6];

- In the first phase high-speed air is circulated around the pieces to eliminate the formation of a saturated air boundary layer on the surface that could lead to condensation [6].

The flow of hot air is very turbulent, in order to try to reach the areas that are normally "in the shadows" of the piece (internal surfaces) through the casting holes. This air is often inverted, rendering the fluid distribution as uniform as possible. This treatment usually lasts 1.5 to 2 hours [6].

- The second phase, that is the vacuum phase, is carried out in a controlled depression and temperature room. Heating is affected by irradiation [6].

The vacuum time varies from two to three hours depending on the type of article to be dried and its body. The main advantage of vacuum is the reduction in the water evaporation temperature and the faster drying speed [7].

*In microwave and hot air dryers*, the piece is heated homogeneously through its entire thickness, thanks to the use of microwaves. The air evacuates the evaporated water and, if heated, improves the technical performance of the machines. Drying times are considerably reduced compared to traditional systems and may vary from 3 to 4 hours depending on the type of sanitary-ware appliance, its shape and the body [3].

## 2.4 PROBLEMS ASSOCIATED WITH THE DRYING PROCESS

Undoubtedly many defects in ceramic articles can be attributed to incorrect methods of drying. The faults, in some cases, are not apparent until the ware has been fired, when they have been accentuated by changes brought about by the heat treatment [13].

In general, the defects, which arise as the result of bad drying methods, are of four types:

*(a)* Those due to differential shrinkage and the consequent induced stresses in the ware

*(b)* Those due to the migration of the finer particles of the body by moisture transference to the drying surface

*(c)* Those which are due to the accentuation of differential stresses arising initially in the forming process

*(d)* Those due to too rapid volatilization of water within the body of the article, which is unable to escape through the fine, pore structure at a sufficiently rapid rate [13].

During drying, a liquid concentration gradient at the beginning of the falling rate period may cause *differential shrinkage* and a tensile stress in the surface. Unfired ceramics are normally quite weak and can sustain stresses of only a few 100 kPa without deformation or local fracture. *Surface cracks* may form when material near the surface becomes brittle and the differential shrinkage produces a stress that exceeds the tensile strength. Differential shrinkage may be produced by gradients in particle size and at junctures between differentially oriented particles of high aspect ratio [8].

Cracks develop along inherent lines of weakness, which in pressed ware are at right angles to the direction at which pressure is applied [13]. Differential shrinkage due to differential liquid content when the product was formed or a differential drying rate across the surface of the product may also produce warping as seen in Figure 2.6.

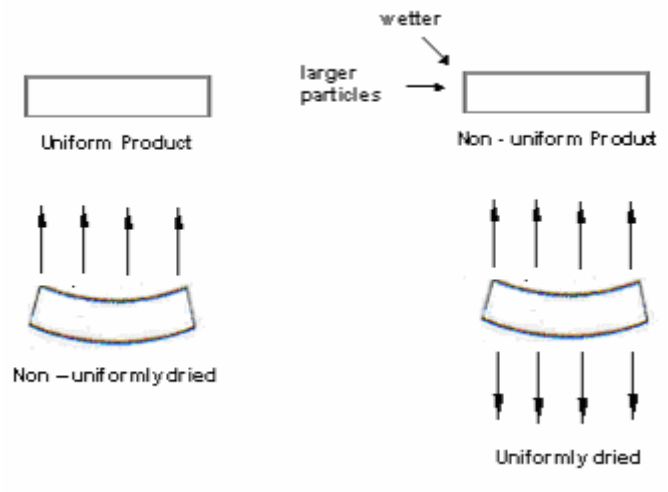



**Figure 2.6.** Shape distortion produced by non-uniform drying shrinkage [8]

*Warping* is caused by stresses accompanying non-symmetrical shrinkage, which produces plastic elongation in regions with a lower shrinkage rate. Increasing the uniformity of drying and reducing the average drying shrinkage of the body reduces warping. The tendency for warping may be increased by uniform external films or coatings, particle orientation, or binder migration, which produce a non – uniform surface permeability and by non-uniformities in the circulation or temperature of drying air due to setting patterns and supporting hardware. Ideally drying should occur in an isotropic material and all shrinkage should end before entering the falling rate period [7].

Many faults normally attributed to incorrect firing may have their origin in incorrect drying practice. *Dunting* in glazed ware is often due to hair – cracks that have developed at the edges of the body during drying. This is a not too

uncommon phenomenon in glaze terra cotta, which has been dried on hot floors. The dunting in such cases is limited to one edge or face that has been dried too rapidly [17].

*Improper drying of ware after the application of the glaze slip may also cause shelling*. Such faults are easily remedied by the correct control of the consistency of the glaze slip, but the defect is apparent after drying and should be clearly differentiated from true shelling, which is due to the incorrect balance between the thermal expansion of the glaze and the body and is appeared only after firing [6].

When drying involves the migration of water from the interior of the article to the surface, fine particled material may also be transported and deposited in the surface layers. This leads to *skin – formation* and an alteration in physical and sometimes chemical properties of the outside layers relative to the internal areas [1].

## 2.5 SUCCESSFUL DRYING SANITARYWARE

The fundamental focus of drying during the constant rate stage is to slow the evaporation from the surface of the ware while increasing the flow of water from the ware's interior; the fact that the capillary flow rate to the surface of the ware is proportional to $K_p / \eta_l$ (where $K_p$ is the permeability of the ware and $\eta_l$ is the viscosity of the water) suggests that there are two ways to increase the water transport rate to the surface. The body could be reformulated to increase the ware's porosity, thus providing the water with easy access to the surface. But this method is impractical since the drying process should not govern the slip making process. The more practical way of increasing transport to the surface is to lower the viscosity of the water. This is accomplished by increasing the temperature. As temperature increases, water will migrate more rapidly to the surface. But this increase in temperature would also elevate the evaporation rate. A practical solution is to heat the ware in humid air. The humid air retards the evaporation

from the surface, allowing the water inside the ware to absorb the heat energy and to decrease in viscosity [4].

An important concept of the drying process to understand is the critical shrinkage point. This is the point when volumetric shrinkage of the ware ceases. When the critical shrinkage point has been reached, the drying rate slows. Because the particles in the ware are in a fixed position, the temperature can be gradually increased while the humidity can be gradually decreased. This will facilitate drying after the evaporation surface has receded into the ware [18].

Conventional drying methods offer little room for acceleration. A remedy, however, is at hand in the form of a familiar technology: the microwave. Drying with microwave technology is both fast and gentle on the product. These qualities assure it a place among drying techniques of the future [19].

Modern drying methods are expected to reduce breakage rates, repeat the drying process with perfect precision every time, and document each drying process to permit the immediate tracing of all potential disturbances throughout the entire procedure [2].

# CHAPTER 3

# MATERIALS AND METHODS

## 3.1 SAMPLE PREPARATION

### 3.1.1 RAW MATERIALS

The raw materials used in this study were industrial grade chemicals; feldspar, quartz, clay, and kaolinite. Feldspar and quartz were used as the non-plastic raw materials and clay and kaolinite were used as the plastic raw materials. The chemicals were obtained from the slip preparation shop of Eczacıbaşı Vitra Ceramic Group, at Bozüyük Plants.

The characteristics of the raw materials are given in Table 3.1.

**Table 3.1** The characteristics of the raw materials

| Raw Material | Size Distribution (μm) | Purity (%) |
|---|---|---|
| Feldspar | 2-25 | 85 - 96 |
| Quartz | 2.4 - 32 | 98 |
| Clay | 0.05-9 | 85 - 96 |
| Kaolinite | 0.5-10 | 85 - 96 |

As seen from Table 3.1, the particle size of feldspar changed from 2.0 to 25 μm and that of quartz changed from 2.4 to 32 μm, while for clay, it varied from 0.05 to 9 μm and for kaolinite it varied from 0.5 to 10 μm. As far as the purity of the raw materials was concerned, it changed from 85 to 96 % for feldspar, clay and kaolinite and it was almost 98 % for quartz.

### 3.1.2 CASTING SLIP PREPARATION

On the basis of investigation on the formation of casting slips, a recipe designated as $R_p$ throughout this study was prepared from the above-mentioned raw materials. It is currently being used as the casting slips for sanitary wares primarily on the production of lavatories and toilet closets in Eczacıbaşı Vitra Ceramic Group, at Bozüyük Plants. The ingredients and amount of the chemicals in weight percentage (wt %) of the recipe are presented in Table 3.2. It contains 50 wt % plastic and 50 wt % non-plastic raw materials. In addition to this recipe, two other recipes designated as $R_1$ and $R_2$ in Table 3.2 were prepared to investigate the effect of non-plastic raw material contents on drying rate. The non-plastic raw material contents in the slips for $R_1$ and $R_2$ were 45 wt % and 55 wt %, respectively.

**Table 3.2** Ingredients and recipes of the slips prepared

| Raw Materials | Chemical Formula | Amounts (wt %) | | |
|---|---|---|---|---|
| | | $R_p$ | $R_1$ | $R_2$ |
| Feldspar | $KAlSi_3O_8$ | 31 | 28 | 34 |
| Quartz | $[98SiO_2.2Al_2O_3]$* | 19 | 17 | 21 |
| Clay | $Mg_2Al_{10}Si_{24}(OH)_{12}$ | 32 | 35 | 29 |
| Kaolinite | $Al_2Si_2O_5(OH)_4$ | 18 | 20 | 16 |

* Weight percent composition.

The starting materials were carefully weighed (±0.01 g) to their proper amount in an analytical balance to form the batches. Coarse raw materials; i.e., feldspar and quartz, were wet ground in a grinder to obtain homogeneous slips. $Al_2O_3$ balls were used as the grinding medium. A small amount (0.2 wt %) of barium carbonate ($BaCO_3$) was added to all mixtures during grinding in order to halt fired surface scumming. $BaCO_3$ is slightly soluble in water and provides $Ba^{++}$ ions to link with $SO4^{--}$ ions in water to form barium sulfate ($BaSO_4$). This new sulfate molecular form is much less soluble, so it stays internal, rather than migrating to the surface during drying.

Then, the mixture was taken to the blunger in order to add clay and kaolinite. Before adding the clay and kaolinite into the blunger, water was added in accordance with the liter weight of the prepared slip. The amount of water to be added to the mixture can be calculated by the following equation;

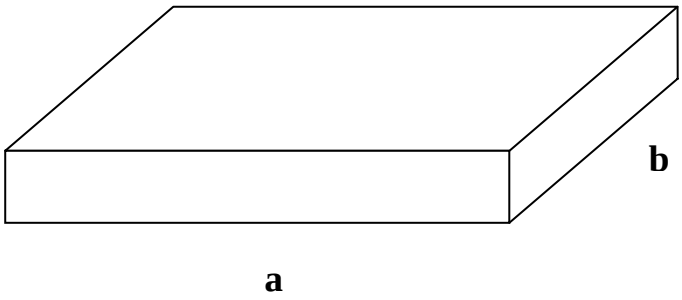
$$m_{water} = (S.G - B.D) / (S.G - 1) \quad (3.1)$$

Where, S.G is the specific gravity of casting slip, which was taken as 2.65

B.D is the bulk density.

A small amount (0.18 wt %) of sodium silicate ($Na_2SiO_3$) was added to all recipes during blunging in order to form a syrupy liquid. Sodium silicate is used as a major defflocculant in preparing slip. Clay particles are chemically charged and allowed to float in suspension in far less water than they would normally. This has the effect that the slip will be fairly viscous after standing in the bucket for a while.

Three slip plates were cast from each recepies to get sufficient number of samples in each stage of the experiments. The dimensions (length x width x depth) of the plates were approximately 135x62x16 mm, respectively. Schematic representation of the plates is illustrated in Figure 3.1.




a = 135 mm
b = 62 mm
c = 16 mm

**Figure 3.1.** Schematic representation of the experimental slip plates

The color of the slip plates were dark green when the plates were taken out of the plaster moulds, but the color turned to gray as drying commenced. The plates were weighed immediately after casting ($W_1$) and located in casting shop environment. The temperature and relative humidity of the casting shop were approximately 31 $^{o}$C and 60 %, respectively.

### 3.1.3 DESCRIPTION OF THE DRYER

The type of dryer, which was produced by Thermic Designs Ltd., used in the experiments at Bozüyük Plant is the batch loaded chamber dryer for cast sanitary ware pieces, having the capability of drying the products from an initial moisture content of almost 19.5 % wet basis down to 0.5 %, or less moisture content. Natural gas is used as the heating medium with the addition of waste heat from kilns.

The dimensions (length x width x depth) of the drier are 12600x4900x2800 mm and the number of 2-level ware trucks per drying cycle is 12. A humidity controlled environment is provided to protect the wares during the critical early phase of the drying cycle, thus allowing the ware to be heated gently without excessive evaporation.

## 3.2 TESTING

### 3.2.1 MEASUREMENT OF WEIGHT LOSS

In order to measure the weight loss of the slip plates during drying, an SBA 51 type of Scaltec scale with an accuracy of 0.01 g was employed. After casting the plates, they were weighed directly ($W_1$). The plates were weighed again ($W_2$) before putting into the dryer. Then, the plates were placed into the dryer having a certain atmosphere for a certain time depending on the experiment. Upon completion of the experiment, the plates were weighed once again ($W_3$) and the results were recorded. The weight loss of the plates was calculated by using the following formula;

$$\% \text{ Weight Loss} = [(W_1) - (W_3) / (W_1)] \times 100 \quad (3.2)$$

### 3.2.2 CALCULATION OF MOISTURE LEVEL

Initial moisture level was defined by weighing three plates ($W_i$) that were prepared by certain sanitary ware slurry in accordance with a known recipe. Then the plates were weighed after drying ($W_f$) at 110 $^oC$ for 15 hours to ensure that the plates were brought to 0 % moisture level. Consequently, the difference between the values $W_i$ and $W_f$ gives the initial moisture level as the plates were taken out of the moulds as given in Equation 3.3.

$$\% \text{ Initial Moisture Level} = [(W_i - W_f) / W_i] \times 100 \quad (3.3)$$

In order to calculate the percent moisture level of the ware after drying, the following formula was used;

$$\% \text{ Moisture Level} = [\% \text{ Initial Moisture Level} - \% \text{ Weight Loss}] \quad (3.4)$$

### 3.2.3 MEASUREMENT OF DRYING SHRINKAGE

Drying shrinkage of the slip plates were measured by a ruler. In order to make the measurement, two signs of 100 mm – long were marked onto the plates diagonally. Then the samples were dried and the signs were measured with a ruler again ($L_1$ and $L_2$) and the shrinkage was calculated by using the following equations;

$$\text{Linear Shrinkage 1} = 100 - L_1 \quad (3.5)$$

$$\text{Linear Shrinkage 2} = 100 - L_2 \quad (3.6)$$

$$\text{Total Linear Shrinkage} = [(100 - L_1) + (100 - L_2)] / 2 \quad (3.7)$$

### 3.2.4 MEASUREMENT OF VISCOSITY

Viscosity is the measurement of the consistency of a slip, which gives a numerical value to its resistance to flow. The instrument used for measuring viscosity is the Brookfield, based on the nature of slip rheology itself. The absolute viscosity of the casting slips was measured at 28 $^{o}$C using a Brookfield Cone and Plate Viscometer. The viscosity data was recorded on an experiment data sheet.

The units used are ‘poise’. The Gallenkamp Torsion Viscometer is widely used in industry and it gives acceptable results for routine control in the factory environment.

## 3.3 DRYING PARAMETERS INVESTIGATED

### 3.3.1 EFFECT OF RESIDENCE TIME FOR THE EXPERIMENTAL SLIP PLATES PRIOR TO LOCATING TO THE DRYER

Three different places in the casting shop were chosen and three experimental slip plates for each place were used and their initial weights were noted just after their casting. Weighing was carried out by 1 hour-intervals as the plates dried. In each time interval, percent weight loss and percent shrinkage was measured and the values found were compared in accordance with the dryness of the area where measurements were produced were performed. The temperature of casting shop, where the measurements were carried out was between 29 $^{o}$C and 31 $^{o}$C and the value of the relative humidity was between 55 – 60 %.

The residence time for all the experiments carried out for the investigation of drying temperature, drying time, percent relative humidity and the non- plastic content in the slip recipe was applied as 5 hours.

### 3.3.2 EFFECT OF TEMPERATURE

Drying temperature was started from nearly 33 $^{o}$C, which was the temperature of the drying chamber before placing the green wares and ended at four different temperature set values. That is, 80,90, 100 and 110 $^{o}$ C in the last drying phase. The drying cycle was broken down into four phases with the requirements for heat and air following the evaporation curve of the products.

In the first phase of the drying cycle, temperature was raised from 33 $^{o}$C to 45 $^{o}$C with a rate of 0.1 $^{o}$C/min. In phase two, temperature was raised from 45 $^{o}$C to 50 $^{o}$C with a rate of 0.08 $^{o}$C/min. In phase three, the temperature was raised from 50 $^{o}$C to the final temperature set point with a rate changing from 0.5 to 1 $^{o}$C/min. Up to phase three, since the critical moisture content was reached and the shrinkage

essentially stops, higher temperature set points could be applied in the third phase. In phase four, the final temperature was held almost constant (± 3 $^{o}$C) until the end of the cycle.

### 3.3.3 EFFECT OF DRYING TIME

Total drying cycle for the slip plates was tried to decrease by lowering the drying time from 10 through 7 hours in the last drying phase provided that the temperature in the last phase was kept at 100 $^{o}$ C (± 3 $^{o}$C).

### 3.3.4 EFFECT OF RELATIVE HUMIDITY

In the existing process, 65 % relative humidity level at the beginning of the drying cycle is used. In this part of the case study, relative humidity value was applied as 60 %, 65 %, 70 % and 75 % and the changes were observed. To ensure that the required high relative humidity levels are maintained without excessive evaporation from the slip plates, a direct steam injection humidifier was used to maintain the relative humidity set point and the data were transmitted to a recorder for each half hour.

### 3.3.5 EFFECT OF NON-PLASTICS

The effect of non-plastics content in the slip recipe to the drying rate was studied. In order to see the effect of non-plastic usage in the slip recipe, the total amount of feldspar and quartz contents was changed in the prepared batch and two different slip recipes were prepared.

## 3.4 EXPERIMENTAL FLOWCHART

Schematic representation of the experimental procedure for determining the drying rate of experimental slip plates is shown in Figure 3.2.

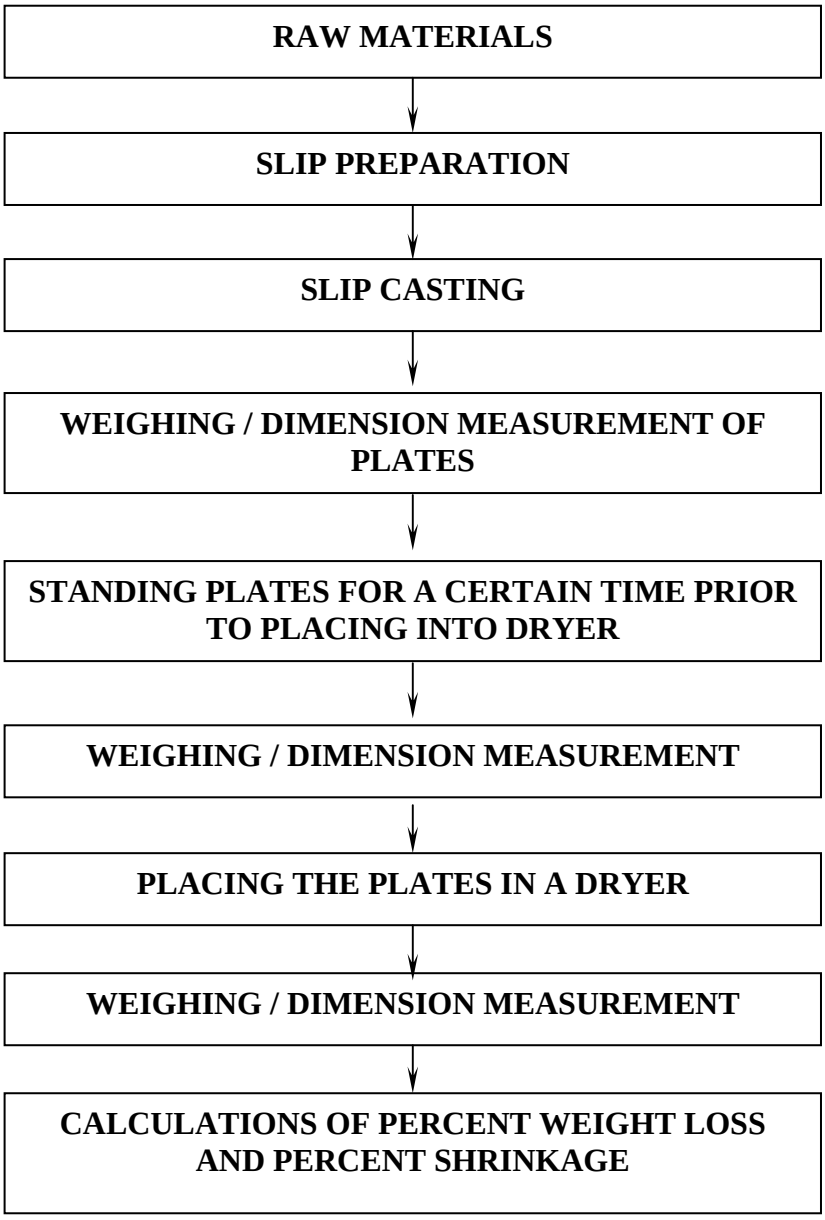



**Figure 3.2.** Flow chart of experimental procedure

# CHAPTER 4

# RESULTS AND DISCUSSION

## 4.1 GENERAL

Data obtained during experimental studies of the thesis work are presented and discussed in this section.

The slip cast samples were prepared in plate form according to the experimental procedure as described in Section 3.1.2. Then the effects of different drying parameters; that is, residence time prior to placing samples into the dryer, temperature, time and relative humidity, which is defined as the ratio at actual mass of water vapour in a given air volume to mass of water vapour required to saturate this volume, in the dryer as well as slip recipes on drying rate and drying shrinkage were investigated.

The tests were conducted as described in Section 3.2 to obtain data on the drying properties of the samples. Data on drying rate and drying shrinkage were gathered through quantitative measurements and calculations.

## 4.2 RHEOLOGICAL PROPERTIES OF THE SLIPS

The characteristics of the raw materials and the rheology of the casting slip have great influence on drying process of sanitary wares. Rheological properties of the slips were determined at 28 $^{o}$C and the results are given in Table 4.1.

**Table 4.1.** The rheological properties of the slips prepared in this study

| **Rheological Parameters** | **Slips Prepared*** | | |
|---|---|---|---|
| | **$R_p$** | **$R_1$** | **$R_2$** |
| Bulk Density (g / $cm^3$) | 1.805 | 1.805 | 1.805 |
| Torsion 1 ( $^o$ ) | 316 | 313 | 315 |
| Torsion 2 ( $^o$ ) | 208 | 206 | 208 |
| Thixotropy | 108 | 107 | 107 |
| Viscosity (cP) | 560 | 540 | 550 |

* Ingredients and recipes are given in Table 3.2.

The most important criterion to determine the casting behavior of a slip and to identify the casting problems is the value of thixotropy. If the thixotropy of a casting slip is too high, soft and high moisture casts are formed. If the thixotropy is too low, hard and low moisture casts are formed [25]. When a comparison was made among the slips prepared, it can be said that all the slips have more or less the same casting behavior; only the slip prepared by the use of $R_p$ was a little bit softer.

## 4.3 RESIDENCE TIME OF THE PLATES PREPARED WITH THE RECIPE $R_P$ PRIOR TO PLACING THEM INTO THE DRYER

Three slip cast plates prepared from the recipe Rp were kept in three different regions in the casting shop to give off their surface moisture prior to placing them in the dryer. The region where the plates are located is commonly known as greenline. The time elapsed during this period is defined as residence time. The temperature and relative humidity in the greenline were approximately 31 $^o$C and 60 %, respectively.

Time spent on the greenline serves to protect the ware from drafts in the open shop, while allowing moisture gradients in the recently cast wares to homogenize. The ware also begins to dry gently as the moisture is reduced at the end of the greenline, where the wares are loaded into the drying chamber [5].

Residence time after casting was increased up to 24 hours to understand the effect of residence time on drying rate. Results gathered for percent weight loss, percent moisture and percent shrinkage of the three plates prepared from the same recipe were tabulated in Appendix A.

Results obtained for the three plates agree with each other. For a given residence time, the difference between the values of the percent weight loss, percent moisture and percent shrinkage of the plates was not significant.

As seen from Table A1, the maximum difference in percent weight loss from one plate to another for a given residence time period was 0.5 %. The percent weight loss after 24-hour residence time for plate 1, plate 2 and plate 3 were 16.6 %, 16.7 % and 16.8 %, respectively. The average percent weight loss for the three plates after 24-hour residence time was 16.7 %.

As seen from Table A2, the maximum difference in percent moisture from one plate to another for a given residence time period was 0.3%. The percent moisture after 24-hour residence time for plate 1, plate 2 and plate 3 were 1.9 %, 1.8 % and 1.7 %, respectively. The average percent moisture for the three plates after 24-hour residence time was 1.8 %.

As seen from Table A3, the maximum difference in percent shrinkage from one plate to another for a given residence time period was at most 0.2%. The percent shrinkage for all of the plates and the average percent shrinkage for the three plates after 24-hour residence time were 3.0 %.

The results revealed that experimental test conditions were practically uniform in the casting shop and did not change much from one region to another.

The variations of percent weight loss and percent shrinkage of all of the plates with residence time prior to placing them into the dryer are shown in Figure 4.1. Percent weight loss increased continuously with increasing residence time up to 24 hours. The increase was more or less linear up to approximately 6 hours and then became nonlinear for all of the plates. Likewise, almost a linear change in percent shrinkage was observed with increasing residence time up to 6 hours where percent shrinkage was 2.93 %. Thereafter percent shrinkage did not change with increasing residence time and stayed constant at 3 %.

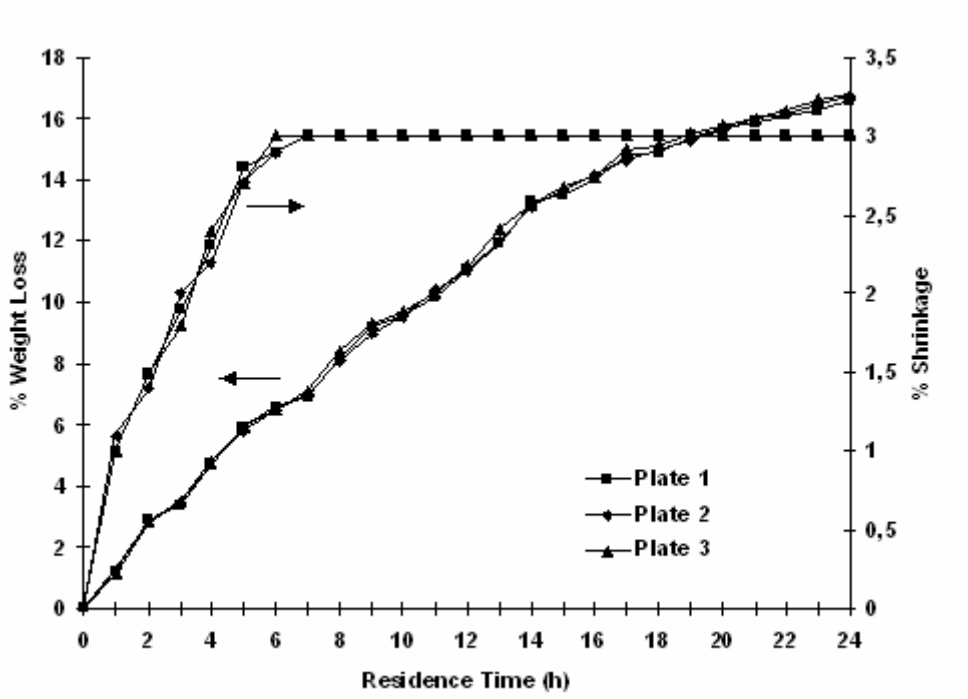



**Figure 4.1.** Variation in percent weight loss and percent shrinkage of the three plates prepared from the recipe Rp with residence time prior to placing them into the dryer

The variation of percent moisture with residence time prior to placing the plates into the dryer is shown in Figure 4.2. Percent moisture decreased continuously with increasing residence time up to 24 hours. The decrease was more or less linear up to approximately 6 hours and then became nonlinear for all of the plates.

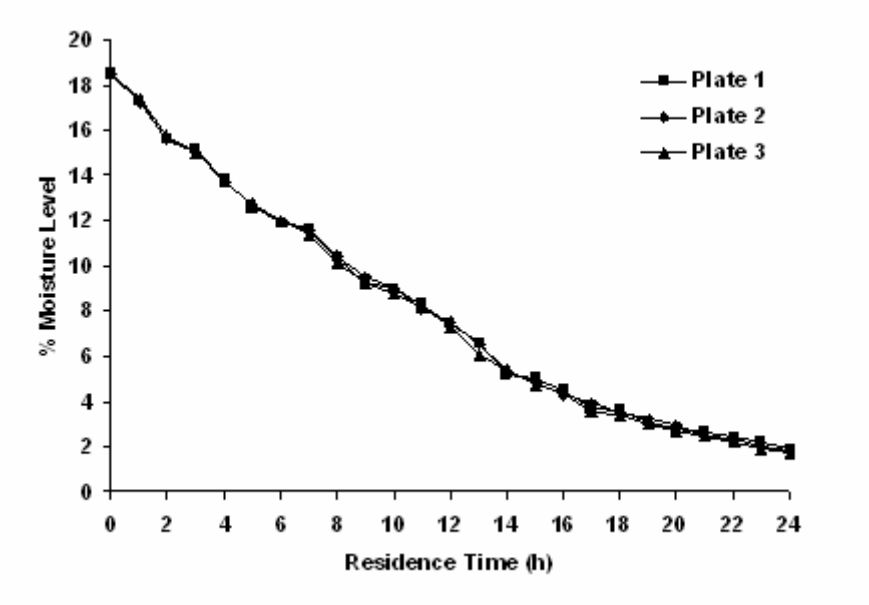



**Figure 4.2.** Variation in percent moisture level of the three plates prepared from the recipe Rp with residence time prior to placing them into the dryer

A decrease in the rate of percent weight loss was observed with increasing residence time. The behavior of percent weight loss, percent moisture and percent shrinkage up to 6 h is mainly due to the constant rate period of drying, that is, the first stage of shrinkage, in which the majority of the shrinkage occurs at a theoretically constant rate. This is the first and most critical stage of drying, where water is actually removed from the green ware body and high shrinkage is observed [3].

Water removal from sanitary ware occurs through evaporation. All evaporation of water occurs at the ware's surface. Consequently, water from inside the piece has to travel through a network of pores to reach the surface [5]. Prior to placing the experimental slip plates into drying chamber, individual grains are separated from each other by a thin film of water. As water leaves the green ware body, the grains move closer together and eventually contact each other. This collapsing of the internal structure results in the body shrinkage as seen in Figure 2.1 [3].

Nevertheless after 6 h, which is the second stage of drying, the rate in percent weight loss started to decrease and percent shrinkage was more or less the same on account of the fact that the structure of the green ware is collapsing as the water film around each ceramic particle is removed [3]. The path for water diffusing from the ware interior to the ware surface is reduced, making it more difficult for the internal water to migrate to the surface for evaporation. Even though the grains are contacting each other, significant amounts of water remain between the grains and inside the pores. During this second stage, that is, first falling rate period of drying, the body continues to lose water at a slower rate through surface evaporation but with very little additional shrinkage [3].

As the particles become closer there is a considerable reduction in the number of openings available for the passage of water and therefore the surface is no longer uniformly covered in liquid. The evaporation phenomenon begins inside the thickness of the ceramic mass with the consequent migration of vapor to the surface. The more the evaporation area moves towards the inside, the greater the reduction in the drying speed [13].

Clausen and Fish [9] conducted a study by using freshly demolded sanitary ware pieces. The temperature and percent relative humidity in the greenline were held constant at 30 $^{o}$C and 80 % R.H., respectively. They found that the residence time of 5 – 7 h was sufficient for the demolded sanitary ware pieces. Although the conditions of the experiment, i.e., temperature and percent relative humidity, were

somewhat different in the study conducted by Clausen and Fish, the obtained results were similar.

## 4.4 EFFECT OF TEMPERATURE

Three slip cast plates prepared from the recipe Rp were placed into the dryer to determine the effect of temperature on the drying rate after a residence time of 5 hours. Data were gathered through the measurements conducted according to the experimental procedure as described in Section 3.2.1 through 3.2.3. The peak temperature used in the dryer was set to 80 $^{o}$C to 110 $^{o}$C. While applying the temperature changes, initial moisture content was kept constant as 18.5 %.

The drying cycle was broken down into four phases with the requirements for heat and air following the evaporation curves of the products as described in Section 3.3.2. Figure 4.3 illustrates a typical drying cycle for a 10-hour drying period.

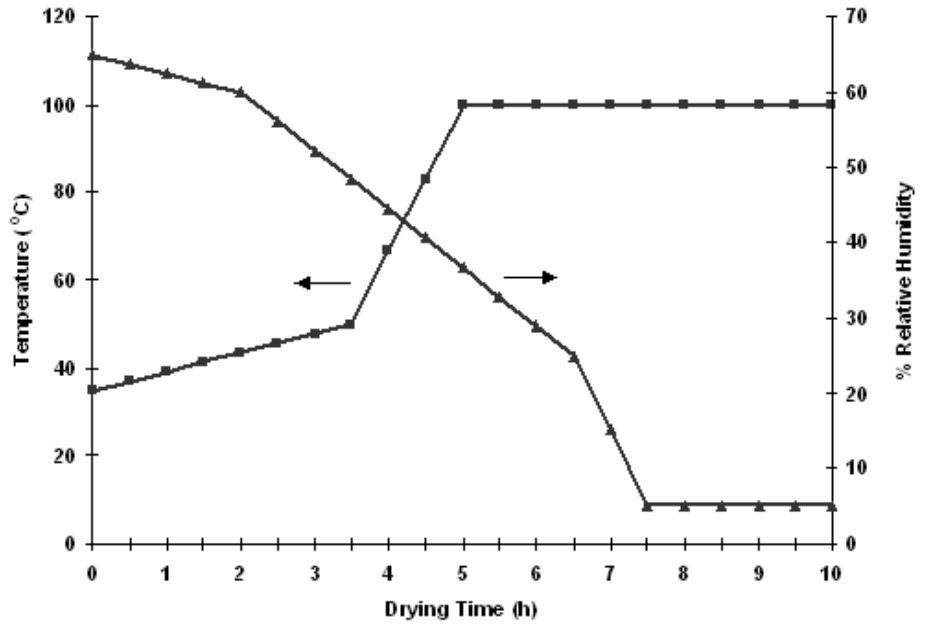



**Figure 4.3.** A typical drying regime for a 10 h drying cycle at 100 $^{o}$C

The drying regimes for 80, 90, 100 and 110 $^{0}$C were illustrated in Figure 4.4. Experimental results are given in Tables 4.2 through 4.5.

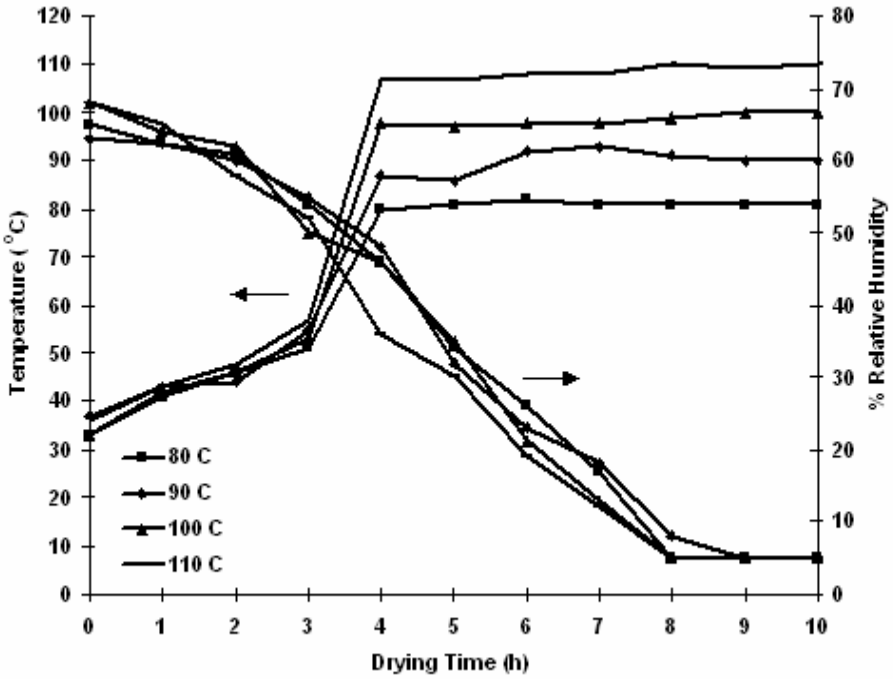



**Figure 4.4.** Drying regimes at 10 hour drying cycle for experimental slip plates at the set points of 80, 90, 100 and 110 $^{o}$C

For the plates dried at 10 h drying cycle at 80 $^{o}$C, the temperature was varied from 33 to 82 $^{o}$C and the percent relative humidity changed from 5 to 65 % as seen in Figure 4.4. From the beginning of the last drying phase, i.e. last 6 hours, the peak temperature was 82 $^{o}$C and it stayed almost constant to the end of the cycle. In the last drying phase, the minimum value of percent relative humidity was 5 % and it stayed constant in the last three hours of the drying cycle.

**Table 4.2.** Percent average weight loss, percent average moisture and percent average shrinkage values for the experimental slip plates at 80 $^{o}$C

| Plate Number | % Weight Loss | | % Moisture | | | % Shrinkage | |
|---|---|---|---|---|---|---|---|
| | Before Placing into the Dryer | After Taking from the Dryer | Just After Casting | Before Placing into the Dryer | After Taking from the Dryer | Before Placing into the Dryer | After Taking from the Dryer |
| **1** | 4.45 | 17.29 | 18.50 | 14.05 | 1.21 | 2.85 | 2.90 |
| **2** | 5.01 | 17.42 | 18.50 | 13.49 | 1.08 | 3.00 | 2.93 |
| **3** | 3.28 | 17.52 | 18.50 | 15.22 | 0.95 | 2.74 | 2.93 |

When a comparison was made among the three plates, it was seen that there was slight deviation from one plate to another in testing parameters. That is, the changes in percent weight loss, percent moisture and percent shrinkage were 0.23 %, 0.26 % and 0.03 %, respectively. It is probably due to the casting behavior of the plaster mould or the difference in holding time of the plates in the mould before demolding. It may also because of the location of the slip plates in the drying chamber.

From the point of view of testing parameters, it was observed that while percent weight loss increased, percent residual moisture decreased and percent shrinkage increased in the same manner for all plates as expected.

For the plates dried at 10 h drying cycle at 90 $^{o}$C as seen in Figure 4.4, the temperature was varied from 37 to 93 $^{o}$C and the percent relative humidity changed from 5 to 63 %. From the beginning of the last drying phase, i.e. last 6 hours, the peak temperature was approximately 90 $^{o}$C and it stayed almost constant to the end of the cycle. In the last drying phase, the minimum value of percent relative humidity was 5 % and it stayed constant in the last two hours of the drying cycle.

**Table 4.3.** Percent average weight loss, percent average moisture and percent average shrinkage values for the experimental slip plates at 90 $^{o}$C

| Plate Number | % Weight Loss | | % Moisture | | | % Shrinkage | |
|---|---|---|---|---|---|---|---|
| | Before Placing into the Dryer | After Taking from the Dryer | Just After Casting | Before Placing into the Dryer | After Taking from the Dryer | Before Placing into the Dryer | After Taking from the Dryer |
| **1** | 1.94 | 17.68 | 18.50 | 16.56 | 0.82 | 2.90 | 3.03 |
| **2** | 5.64 | 17.79 | 18.50 | 12.86 | 0.71 | 2.97 | 3.05 |
| **3** | 3.62 | 17.38 | 18.50 | 14.88 | 1.12 | 2.93 | 3.04 |

By the same token, there was again slight difference in testing parameters. That is, the changes in percent weight loss, percent moisture and percent shrinkage were 0.41 %, 0.41 % and 0.02 %, respectively. It is probably in view of the casting conditions of the slip plates or the placement of the plates in the dryer. As far as the testing parameters are concerned, the results revealed that as percent weight loss increased, percent residual moisture decreased and percent shrinkage increased in the same way for all plates.

When the results found at 90 $^{o}$C were compared to that of 80 $^{o}$C, as temperature increased by 10 $^{o}$C, percent weight loss and percent shrinkage increased by 0.21 % and 0.12 %, respectively on the average, nevertheless percent residual moisture decreased by 0.2 %.

For the plates dried at 10 h drying cycle at 100 $^{o}$C as seen in Figure 4.4, the temperature was varied from 35 to 100 $^{o}$C and the percent relative humidity changed from 5 to 68 %. From the beginning of the last drying phase, i.e. last 6 hours, the peak temperature was 100 $^{o}$C and it stayed almost constant to the end of

the cycle. In the last drying phase, the minimum value of percent relative humidity was 5 % and it stayed constant in the last three hours of the drying cycle.

**Table 4.4.** Percent average weight loss, percent average moisture and percent average shrinkage values for the experimental slip plates at 100 $^{o}$C

| Plate Number | % Weight Loss | | % Moisture | | | % Shrinkage | |
|---|---|---|---|---|---|---|---|
| | Before Placing into the Dryer | After Taking from the Dryer | Just After Casting | Before Placing into the Dryer | After Taking from the Dryer | Before Placing into the Dryer | After Taking from the Dryer |
| **1** | 3.64 | 17.93 | 18.50 | 14.86 | 0.57 | 2.92 | 3.08 |
| **2** | 4.58 | 18.02 | 18.50 | 13.92 | 0.48 | 2.95 | 3.11 |
| **3** | 3.58 | 17.88 | 18.50 | 14.92 | 0.62 | 2.90 | 3.08 |

In a similar way, the results found at 100 $^{o}$C were more or less the same for the three plates. The changes in percent weight loss, percent moisture and percent shrinkage were 0.14 %, 0.14 % and 0.03 %, respectively. The results showed that as percent weight loss increased, percent residual moisture decreased and percent shrinkage increased in a similar way.

When the results found at 100 $^{o}$C were compared to that of 90 $^{o}$C, an increment of 10 $^{o}$C in temperature gave rise to an increment of 0.35 % in percent weight loss, 0.05 % in percent shrinkage and a decrease of 0.33 % in percent residual moisture content on the average.

For the plates dried at 10 h drying cycle at 110 $^{o}$C as seen in Figure 4.4, the temperature was varied from 36 to 110 $^{o}$C and the percent relative humidity changed from 5 to 68 %. From the beginning of the last drying phase, i.e. last 6 hours, the peak temperature was 110 $^{o}$C and it stayed almost constant to the end of

the cycle. In the last drying phase, the minimum value of percent relative humidity was 5 % and it stayed constant in the last three hours of the drying cycle.

**Table 4.5.** Percent average weight loss, percent average moisture and percent average shrinkage values for the experimental slip plates at 110 $^{o}$C

| Plate Number | % Weight Loss | | % Moisture | | | % Shrinkage | |
|---|---|---|---|---|---|---|---|
| | Before Placing into the Dryer | After Taking from the Dryer | Just After Casting | Before Placing into the Dryer | After Taking from the Dryer | Before Placing into the Dryer | After Taking from the Dryer |
| **1** | 3.79 | 17.96 | 18.50 | 14.71 | 0.54 | 2.91 | 3.00 |
| **2** | 4.52 | 18.08 | 18.50 | 13.98 | 0.42 | 2.94 | 3.13 |
| **3** | 4.21 | 18.10 | 18.50 | 14.29 | 0.40 | 2.93 | 3.20 |

In this part of the experiment, nearly the same results were obtained. The changes in percent weight loss, percent moisture and percent shrinkage were 0.14 %, 0.15 % and 0.20 %, respectively. The results revealed that experimental test conditions were practically uniform and did not change much from one plate to another.

As temperature increased from 100 to 110 $^{o}$C, percent weight loss and percent shrinkage increased by 0.10 % and 0.02 %, respectively on the average, nevertheless percent residual moisture decreased by 0.10 %. It can be seen that the change in the results of testing parameters after reaching 110 $^{o}$C became almost negligible.

**Table 4.6.** Results of percent average weight loss, percent average moisture and percent average shrinkage for experimental slip plates at different temperatures

| Temperature ($^oC$) | % Average Weight Loss | % Average Shrinkage | % Average Moisture |
|---|---|---|---|
| **80** | 17.42 | 2.92 | 1.08 |
| **90** | 17.62 | 3.04 | 0.88 |
| **100** | 17.94 | 3.09 | 0.56 |
| **110** | 18.05 | 3.11 | 0.45 |

The average percent weight loss varied from 17.42 to 18.05 %, average percent moisture varied from 0.45 to 1.08 % and the average percent shrinkage varied from 2.92 to 3.09 for different temperatures as seen in Table 4.6.

The typical temperature vs. percent average weight loss, percent average moisture and percent average shrinkage curves for the plates dried at 10 h drying cycle at different temperatures, i.e. 80, 90, 100 and 110 $^oC$ are shown in Figure 4.5. The average value of percent moisture decreased with increasing temperature. Nevertheless, the average value of percent shrinkage and percent weight loss increased with increasing temperature. If we evaluate the average moisture contents for all temperatures studied, it absolutely seems that the value, 0.56 % found at 100 $^oC$ is reasonable since the percent moisture value should be 0.7 % or less.

When the green ware is dried to moisture content of 0.5 to 1 %, it becomes strong enough for subsequent processing, and after the elimination of residual moisture at 110 – 120 $^oC$, the ware attains its maximum strength. Well-dried ware can withstand a rapid rise in temperature during the initial firing period, and as a result

the productivity of the kilns is improved and the amount of fuel used during firing is reduced [10]. Therefore, the value of 0.7 % moisture level is associated with the firing rate. In Vitra, the total firing period for the glazed wares is 12 h. It had been seen that residual moisture contents greater than 0.7 % gave rise to cracks or even blew the wares apart for 12 h – firing period.

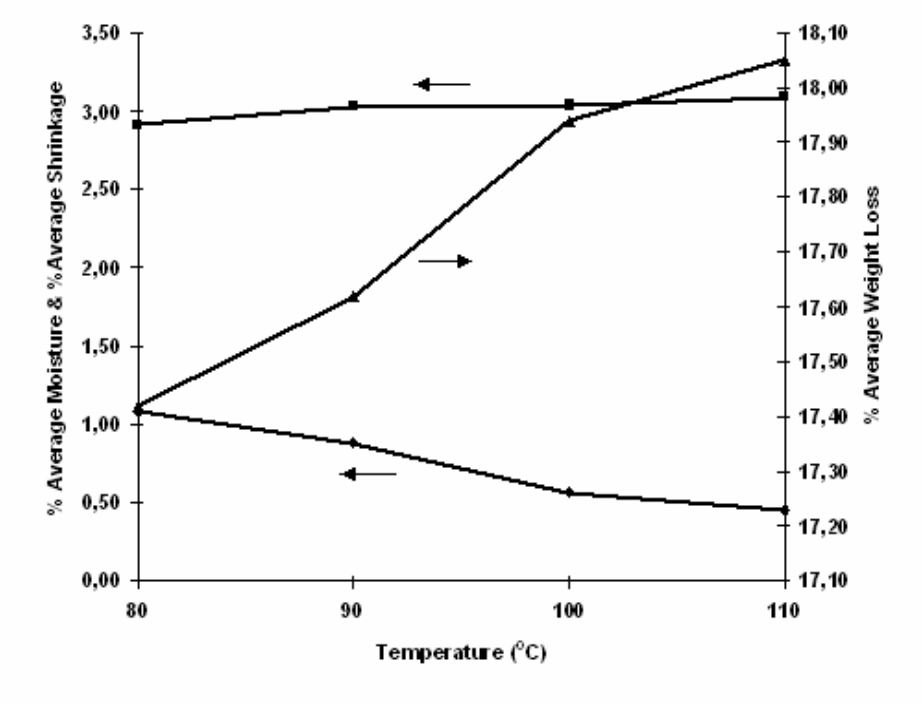



**Figure 4.5.** Variation of percent weight loss, percent moisture and percent shrinkage with temperature for all plates

The drying process can be conveniently divided into two periods; a constant rate period and a falling rate period. During the former, the wet material contains so much water that the surface of the body consists of a continuous film of water. This evaporates at a rate comparable to that of a free water surface and is thus dependent only on the ambient conditions (temperature, velocity and humidity of the air) and the surface area and the temperature of the water film. Once the critical moisture content is reached, however, the continuous film is broken and

the falling period starts. This stage involves two processes; migration of moisture within the material to the surface and removal of the moisture from the surface. The former can occur by three mechanisms; vapor flow, liquid flow and diffusion. The latter is usually only significant when the moisture content drops below the saturation point. Liquid flow dominates with convective drying, where the rate of moisture movement is given by the equation [7];

$$dM / dt = k\, p(C_d - C_w) / l\, \eta \qquad (4.1)$$

where $C_d$ and $C_w$ are the water concentrations at the dry and wet phases of the body respectively, p is the permeability of the body, l is the path length, $\eta$ is the viscosity of water and k is a constant [7].

From this relation, it is evident that to achieve a high rate of water flow and a large moisture gradient or a low water viscosity i.e., a high drying temperature would be required for a body of given permeability and size [7]. As the temperature of the water inside the body increases, the viscosity decreases, allowing the water to migrate to the surface more readily [20].

The rate of transfer of the moisture to the surface of the ware is affected by the properties of the mixture, the size of the capillaries, and the viscosity of the liquid (in the given case water), which declines sharply as the temperature is raised. For example, at 70 $^{o}$C, the viscosity of water is approximately four times less than at 0 $^{o}$C [17]. In this study, at 80 and 90 $^{o}$C, liquid flow is not enough to obtain a suitable moisture level for the bodies. However, after 100 $^{o}$C, the movement of vapor molecules is activated to the desired diffusion rate and optimum values are observed. It should be also pointed out that the surface tension, which is also a force transferring moisture through the capillaries, is also reduced as the temperature rises, but to a lesser degree than the viscosity. In this way, heating the water in the capillaries facilitates its motion towards the surface and reduces the moisture and shrinkage gradients in different areas of the part. Hence, large solid

articles are first heated through in a moist medium. In this case, there is no change of evaporation of large amounts of moisture from the surface before the article heats throughout, or prior to the stage of more intensive circulation of the moisture through the capillaries. Furthermore, variation in the surface tension of water when heated causes thermal diffusion, that is to say, motion of the moisture along the capillaries from the hotter to the cooler surface, thus speeding up the drying [17].

Slevin [5] portrayed the effects of the drying process on a wet, extruded structural clay brick with 15 to 20 % initial water content. He observed that as the rate of drying changed from 0.41 $^{o}$C / min. to 0.83 $^{o}$C / min. in the second stage of drying, the samples did not crack and he found that the optimum temperature for the samples was approximately 120 $^{o}$C. Even though the conditions of the experiment were different in the study conducted by him, the obtained results were similar. It clearly shows that upon changing to more aggressive drying conditions (an increase in temperature and decrease in relative humidity), the rate of water loss and the rate of shrinkage increased.

## 4.5 DRYING TIME

Three slip cast plates prepared from the recipe Rp were placed into the dryer to determine the effect of drying time on the drying rate. Data were gathered through the measurements conducted according to the experimental procedure as described in Section 3.2.1 through 3.2.3. In this part, drying time was decided to be applied as 7, 8, 9 and 10 h. Temperature was kept constant at 100 $^{o}$C since optimum moisture content was obtained at this temperature.

The drying regimes for 7, 8, 9 and 10 h drying cycles at 100 $^{o}$C were illustrated in Figure 4.6. Experimental results are shown in Tables 4.7 through 4.10.

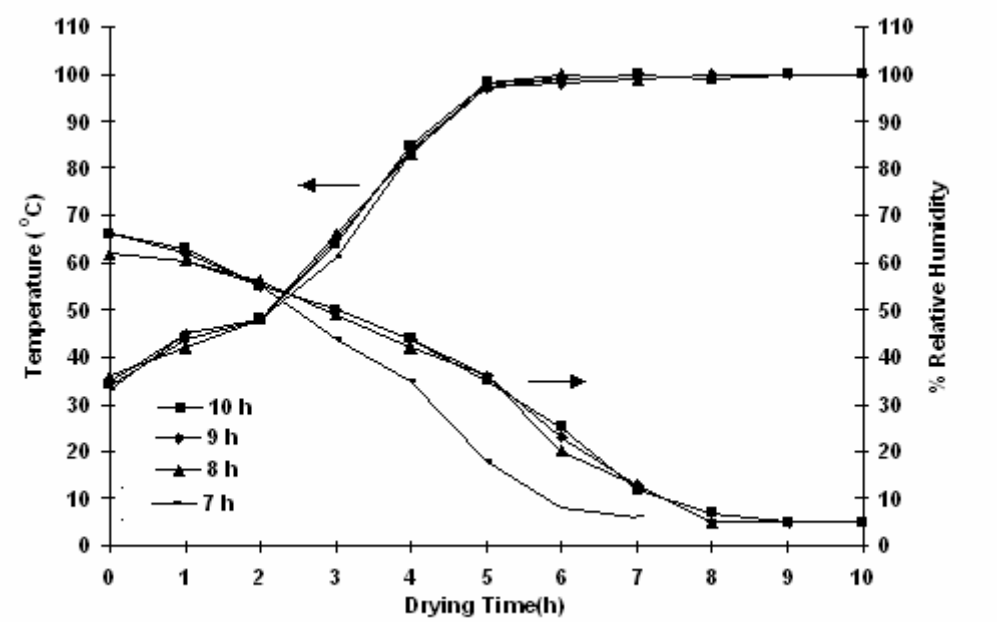



**Figure 4.6.** Drying regimes for 7, 8, 9 and 10 h drying cycles for the experimental slip plates at 100 $^{o}$C

For the plates dried at 100 $^{o}$C for 7 h as seen in Figure 4.6, the temperature was varied from 33 to100 $^{o}$C and the percent relative humidity changed from 6 to 62 %. From the beginning of the last drying phase, i.e. last 4 h, the peak temperature was 100 $^{o}$C and it stayed constant to the end of the cycle. In the last drying phase, the minimum value of percent relative humidity was 6 %.

**Table 4.7.** Results of percent weight loss, percent moisture and percent shrinkage for experimental slip plates after applying 7 h drying cycle at 100 $^{o}$C

| Plate Number | % Weight Loss | | % Moisture | | | % Shrinkage | |
|---|---|---|---|---|---|---|---|
| | Before Placing into the Dryer | After Taking from the Dryer | Just After Casting | Before Placing into the Dryer | After Taking from the Dryer | Before Placing into the Dryer | After Taking from the Dryer |
| **1** | 5.61 | 17.76 | 18.50 | 12.89 | 0.74 | 2.82 | 2.90 |
| **2** | 3.10 | 17.69 | 18.50 | 15.40 | 0.81 | 2.68 | 2.87 |
| **3** | 6.34 | 17.73 | 18.50 | 12.16 | 0.77 | 2.94 | 2.90 |

The changes in percent weight loss, percent moisture and percent shrinkage were 0.07 %, 0.07 % and 0.03 %, respectively, which means that there was not a significant deviation in the results from one plate to another. The slight difference in the results may be due to the casting quality that depends on such factors as holding time of the slip in the mould, mould dryness etc. Also, the data may be differed by drying conditions, i.e. placement of the plates in the drying chamber or heat distribution of the drying air.

From the point of view of testing parameters, it was observed that while percent weight loss increased, percent residual moisture decreased and percent shrinkage increased in the same manner for all plates as expected.

For the plates dried at 100 $^{o}$C for 8 h as seen in Figure 4.10, the temperature was varied from 36 to 100 $^{o}$C and the percent relative humidity changed from 5 to 62 % R.H. From the beginning of the last drying phase, i.e. last 4 h, the peak temperature was 100 $^{o}$C and it stayed constant to the end of the cycle. In the last drying phase, the minimum value of percent relative humidity was 5 %.

**Table 4.8.** Results of percent weight loss, percent moisture and percent shrinkage for experimental slip plates after applying 8 h drying cycle at 100 $^{o}$C

| Plate Number | % Weight Loss | | % Moisture | | | % Shrinkage | |
|---|---|---|---|---|---|---|---|
| | Before Placing into the Dryer | After Taking from the Dryer | Just After Casting | Before Placing into the Dryer | After Taking from the Dryer | Before Placing into the Dryer | After Taking from the Dryer |
| **1** | 4.19 | 17.93 | 18.50 | 14.31 | 0.57 | 2.78 | 3.10 |
| **2** | 4.97 | 17.83 | 18.50 | 13.53 | 0.67 | 2.83 | 2.82 |
| **3** | 2.50 | 17.87 | 18.50 | 16.00 | 0.63 | 2.77 | 2.90 |

By the same token, there was again slight difference in testing parameters. That is, the changes in percent weight loss, percent moisture and percent shrinkage were 0.10 %, 0.10 % and 0.28 %, respectively. It is probably in view of the casting and drying conditions. As far as the testing parameters are concerned, the results revealed that as percent weight loss increased, percent residual moisture decreased and percent shrinkage increased in the same way for all plates.

When the results found at 8 h drying cycle were compared to that of 7 h, as time increased by 1 h, percent weight loss and percent shrinkage increased by 0.25 % and 0.06 %, respectively, nevertheless percent residual moisture decreased by 0.15 % on the average.

For the plates dried at 100 $^{o}$C for 9 h as seen in Figure 4.10, the temperature was varied from 35 to 100 $^{o}$C and the percent relative humidity changed from 5 to 66 %. From the beginning of the last drying phase, i.e. last 5 h, the peak temperature was 100 $^{o}$C and it stayed constant to the end of the cycle. In the last drying phase, the minimum value of percent relative humidity was 5 %.

**Table 4.9.** Results of percent weight loss, percent moisture and percent shrinkage for experimental slip plates after applying 9 h drying cycle at 100 $^{o}$C

| Plate Number | % Weight Loss | | % Moisture | | | % Shrinkage | |
|---|---|---|---|---|---|---|---|
| | Before Placing into the Dryer | After Taking from the Dryer | Just After Casting | Before Placing into the Dryer | After Taking from the Dryer | Before Placing into the Dryer | After Taking from the Dryer |
| **1** | 4.57 | 17.88 | 18.50 | 13.93 | 0.62 | 2.81 | 2.87 |
| **2** | 1.97 | 17.97 | 18.50 | 16.53 | 0.54 | 2.68 | 3.10 |
| **3** | 4.55 | 17.91 | 18.50 | 13.95 | 0.59 | 2.79 | 2.95 |

In a similar way, the results found after applying 9 h drying cycle were more or less the same for the three samples. The changes in percent weight loss, percent moisture and percent shrinkage were 0.09 %, 0.08 % and 0.23 %, respectively. The results showed that as percent weight loss increased, percent residual moisture decreased and percent shrinkage increased in a similar way.

When the results found after applying 9 h drying cycle were compared to that of 8 h drying cycle, an increment of 1 h in time gave rise to an increment of 0.04 % in percent weight loss, 0.03 % in percent shrinkage and a decrease of 0.04 % in percent residual moisture content on the average.

For the plates dried at 100 $^{o}$C for 10 h as seen in Figure 4.10, the temperature was varied from 34 to 100 $^{o}$C and the percent relative humidity changed from 5 to 66 %. From the beginning of the last drying phase, i.e. last 5 h, the peak temperature was 100 $^{o}$C and it stayed constant to the end of the cycle. In the last drying phase, the minimum value of percent relative humidity was 5 %.

**Table 4.10.** Results of percent weight loss, percent moisture and percent shrinkage for experimental slip plates after applying 10 h drying cycle at 100 $^{o}$C

| Plate Number | % Weight Loss | | % Moisture | | | % Shrinkage | |
|---|---|---|---|---|---|---|---|
| | Before Placing into the Dryer | After Taking from the Dryer | Just After Casting | Before Placing into the Dryer | After Taking from the Dryer | Before Placing into the Dryer | After Taking from the Dryer |
| **1** | 3.70 | 17.93 | 18.50 | 14.80 | 0.57 | 2.92 | 3.08 |
| **2** | 4.56 | 18.02 | 18.50 | 13.94 | 0.48 | 2.95 | 3.11 |
| **3** | 3.56 | 17.95 | 18.50 | 14.94 | 0.55 | 2.90 | 3.08 |

In this part of the experiment, nearly the same observations were acquired. The changes in percent weight loss, percent moisture and percent shrinkage were again more or less the same, i.e., 0.09 %, 0.09 % and 0.03 %, respectively. The results revealed that experimental test conditions were practically uniform and did not change much from one plate to another.

As time increased from 9 h to 10 h, percent weight loss and percent shrinkage increased by 0.05 % and 0.12 %, respectively on the average, nevertheless percent residual moisture decreased by 0.05 %.

**Table 4.11.** Results of percent average moisture, percent average shrinkage and percent average weight loss for all plates after applying 7, 8, 9 and 10 h drying cycle

| Time (h) | % Average Moisture | % Average Shrinkage | % Average Weight Loss |
|---|---|---|---|
| **7** | 0.77 | 2.89 | 17.73 |
| **8** | 0.62 | 2.94 | 17.88 |
| **9** | 0.58 | 2.97 | 17.92 |
| **10** | 0.53 | 3.09 | 17.97 |

The average percent weight loss varied from 17.73 to 17.97, the average percent moisture varied from 0.53 to 0.77 % and the average percent shrinkage varied from 2.89 to 3.09 for different drying times as seen in Table 4.11.

The variation of the percent average weight loss, percent average moisture and percent average shrinkage as a function of time for the plates dried at 100 $^{o}$C for different drying times, i.e. 7, 8, 9 and 10 h are shown in Figure 4.7. The average value of percent moisture decreased with increasing drying time. On the other hand, the average value of percent shrinkage and percent weight loss increased when drying time increased. It absolutely seems that the value, 0.62 % found at 100 $^{o}$C for 8 h is reasonable due to obtaining a moisture level lower than 0.7 % [10]. This behavior was interpreted as follows:

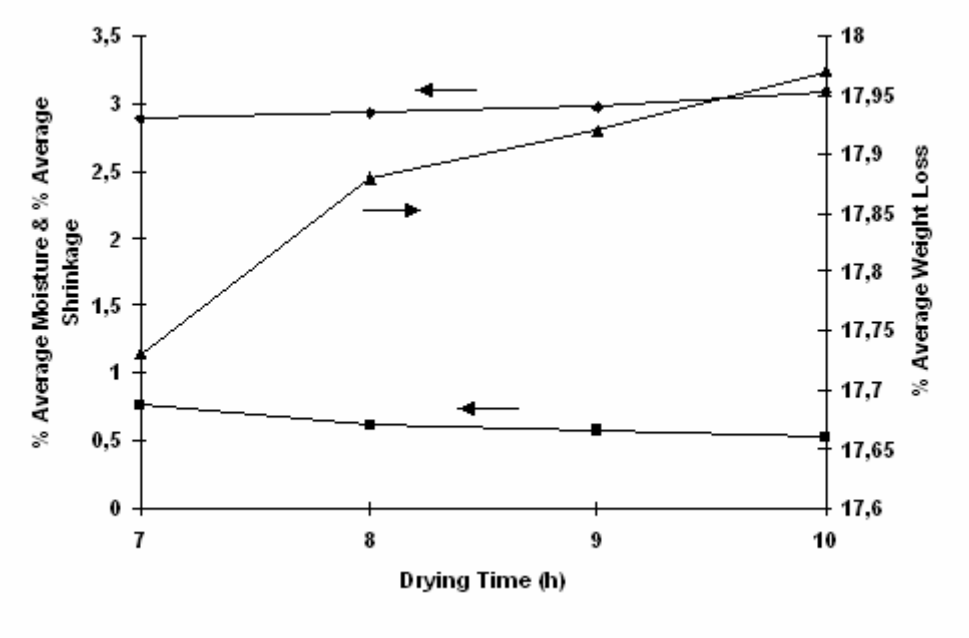



**Figure 4.7.** Variation of percent average weight loss, percent average moisture and percent average shrinkage as a function of drying time at 100 $^{o}$C for the experimental slip plates

To achieve a high rate of water flow, a large moisture gradient would be required [7]. In ceramic products such as sanitary ware, the moisture is transferred from the deeper layers to the surface. Hence the drying period depends on the diffusion of the moisture from the surface into the surrounding medium, and the amount of evaporated moisture as determined by the following equation is a function of the surrounding medium:

$$W = zcF(P_s - P_p)760 / b \qquad (4.2)$$

W is the amount of evaporating water

z is the time

F is the evaporation surface

$P_s$ is the saturated water vapor pressure at the given temperature of the evaporation surface

$P_p$ is the partial water vapor pressure in the surrounding gas medium

b is the barometric pressure of the surrounding atmosphere

c is the coefficient determining the evaporation as a function of the circulation rate of the gases on the surface.

The drying process can be conveniently divided into two periods; a constant rate period and a falling rate period. During the former, the wet material contains so much water that the surface of the body consists of a continuous film of water. Once the critical moisture content is reached, however, the continuous film is broken and the falling period starts. The more the drying time is increased, the greater will be the evaporation within the material to the surface and the more will be the removal of moisture from the surface [10].

Slevin [5] investigated the effect of drying time on the percent weight loss and shrinkage by speeding up the drying schedule. In his work, he concentrated on a series of wet structural clay brick and he saw that the drying time was safely cut by more than half, from 28 to 12 h at 120 $^{o}$C and 70 % R.H. [3].

Based on the result of Slevin [5] and the direct observation in this study, the following explanation may be given: When the elapsed time in the drying chamber increased, both percent weight loss and percent shrinkage increased. Consequently, It seems that the percent average moisture; % 0.62 found at 100 $^{o}$C for 8 h is reasonable in this study.

## 4.6 RELATIVE HUMIDITY IN THE DRYER

Three slip cast plates prepared from the recipe Rp were placed into the dryer to determine the effect of percent relative humidity on the drying rate. Data were gathered through the measurements conducted according to the experimental procedure as described in Section 3.2.1 through 3.2.3.

In Vitra plants, relative humidity at the beginning of the drying cycle is accepted as 65 %. The drying cycles for 60, 65, 70 and 75 % relative humidity were illustrated in Figure 4.8. Experimental results are shown in Tables 4.12 through 4.15.

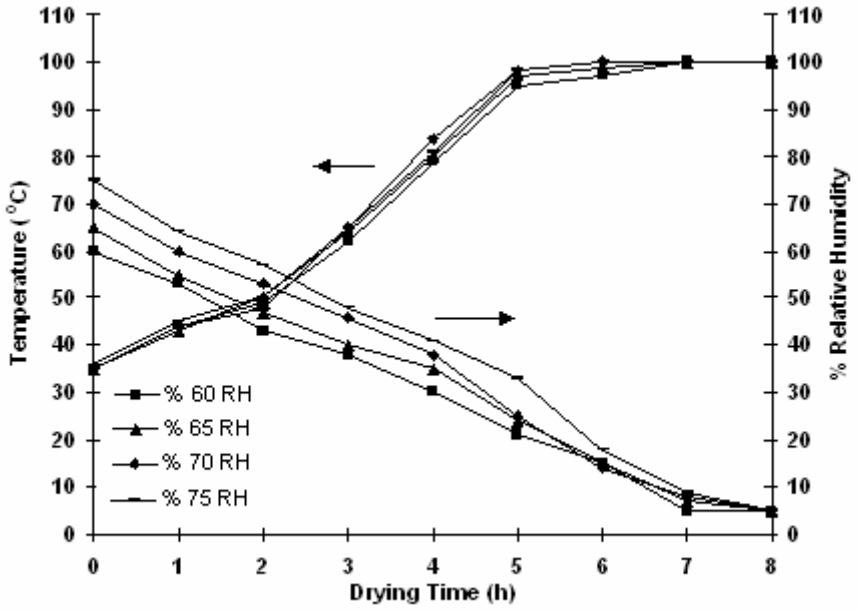



**Figure 4.8.** Drying regimes of the experimental slip plates for 60, 65, 70 and 75 % relative humidity at 8 h and 100 °C drying cycle

For the plates dried for 8 h at 60 % relative humidity as seen in Figure 4.8, the temperature was varied from 35 to 100 $^{o}$C and the percent relative humidity changed from 5 to 60 %. From the beginning of the last drying phase, i.e. last 3 h, the peak temperature was 100 $^{o}$C and it stayed constant to the end of the cycle. In the last drying phase, the minimum value of percent relative humidity was 5 %.

**Table 4.12.** Results of percent weight loss, percent moisture and percent shrinkage values at 60 % relative humidity for the experimental slip plates

| Plate Number | % Weight Loss | | % Moisture | | | % Shrinkage | |
|---|---|---|---|---|---|---|---|
| | Before Placing into the Dryer | After Taking from the Dryer | Just After Casting | Before Placing into the Dryer | After Taking from the Dryer | Before Placing into the Dryer | After Taking from the Dryer |
| **1** | 3.59 | 17.99 | 18.50 | 14.91 | 0.51 | 2.71 | 3.00 |
| **2** | 5.10 | 17.91 | 18.50 | 13.40 | 0.58 | 2.83 | 2.89 |
| **3** | 4.31 | 17.94 | 18.50 | 14.19 | 0.56 | 2.80 | 2.97 |

After applying 60 % relative humidity at the beginning of the drying cycle, the changes in percent weight loss, percent moisture and percent shrinkage were 0.08 %, 0.07 % and 0.03 %, respectively, which means that there was not a significant deviation in data from one plate to another. The slight difference in the results can be associated with the process conditions in casting and the location of the plates in the chamber.

While percent weight loss increased, percent residual moisture decreased and percent shrinkage increased in the same manner for all plates as expected.

For the plates dried for 8 h at 65 % relative humidity as seen in Figure 4.8, the temperature was varied from 35 to 100 $^{o}$C and the percent relative humidity changed from 5 to 65 %. From the beginning of the last drying phase, i.e. last 3 h, the maximum temperature was 100 $^{o}$C and it stayed constant to the end of the cycle. In the last drying phase, the minimum value of percent relative humidity was 5 %.

**Table 4.13.** Results of percent weight loss, percent moisture and percent shrinkage values at 65 % relative humidity for the experimental slip plates

| Plate Number | % Weight Loss | | % Moisture | | | % Shrinkage | |
|---|---|---|---|---|---|---|---|
| | Before Placing into the Dryer | After Taking from the Dryer | Just After Casting | Before Placing into the Dryer | After Taking from the Dryer | Before Placing into the Dryer | After Taking from the Dryer |
| **1** | 4.19 | 17.94 | 18.50 | 14.31 | 0.56 | 2.78 | 3.08 |
| **2** | 4.97 | 17.85 | 18.50 | 13.53 | 0.65 | 2.83 | 2.84 |
| **3** | 2.50 | 17.88 | 18.50 | 16.00 | 0.62 | 2.77 | 2.90 |

When the results were compared among the three plates, it was seen that there was a slight deviation from one plate to another in testing parameters. That is, the changes in percent weight loss, percent moisture and percent shrinkage were 0.09 %, 0.09 % and 0.24 %, respectively.

When the results found at 65 % R.H. were compared to that of 60 % R.H., as relative humidity increased by 5 %, percent weight loss and percent shrinkage decreased by 0.06 % and 0.01 %, respectively on the average, nevertheless percent residual moisture increased by 0.06 %.

For the plates dried for 8 h at 70 % relative humidity as seen in Figure 4.8, the temperature was varied from 35 to 100 $^{o}$C and the percent relative humidity changed from 5 to 70 %. From the beginning of the last drying phase, i.e. last 3 h, the maximum temperature was 100 $^{o}$C and it stayed constant to the end of the cycle. In the last drying phase, the minimum value of percent relative humidity was 5 %.

**Table 4.14.** Results of percent weight loss, percent moisture and percent shrinkage values at 70 % relative humidity for the experimental slip plates

| Plate Number | % Weight Loss | | % Moisture | | | % Shrinkage | |
|---|---|---|---|---|---|---|---|
| | Before Placing into the Dryer | After Taking from the Dryer | Just After Casting | Before Placing into the Dryer | After Taking from the Dryer | Before Placing into the Dryer | After Taking from the Dryer |
| **1** | 2.54 | 17.81 | 18.50 | 15.96 | 0.69 | 2.77 | 2.90 |
| **2** | 3.03 | 17.92 | 18.50 | 15.47 | 0.58 | 2.83 | 3.00 |
| **3** | 5.36 | 17.85 | 18.50 | 13.14 | 0.65 | 2.90 | 2.90 |

The results found in testing parameters at 70 % R.H. were more or less the same for the three plates. The changes in percent weight loss, percent moisture and percent shrinkage were 0.09 %, 0.11 % and 0.10 %, respectively. Likewise, as percent weight loss increased, percent residual moisture decreased and percent shrinkage increased as expected.

When the results found at 70 % R.H. were compared to that of 65 % R.H., an increment of 5 % gave rise to a decrease of 0.03 % in percent weight loss, 0.01 % in percent shrinkage and an increment of 0.03 % in percent residual moisture content on the average.

For the plates dried for 8 h at 75 % relative humidity as seen in Figure 4.12, the temperature was varied from 36 to 100 $^{o}$C and the percent relative humidity changed from 5 to 75 %. From the beginning of the last drying phase, i.e. last 3 h, the peak temperature was 100 $^{o}$C and it stayed constant to the end of the cycle. In the last drying phase, the minimum value of percent relative humidity was 5 %.

**Table 4.15.** Results of percent weight loss, percent moisture and percent shrinkage values at 75 % relative humidity for the experimental slip plates

| Plate Number | % Weight Loss | | % Moisture | | | % Shrinkage | |
|---|---|---|---|---|---|---|---|
| | Before Placing into the Dryer | After Taking from the Dryer | Just After Casting | Before Placing into the Dryer | After Taking from the Dryer | Before Placing into the Dryer | After Taking from the Dryer |
| **1** | 5.21 | 17.86 | 18.50 | 13.29 | 0.64 | 2.72 | 3.00 |
| **2** | 5.79 | 17.79 | 18.50 | 12.71 | 0.71 | 2.90 | 2.90 |
| **3** | 3.90 | 17.70 | 18.50 | 14.60 | 0.68 | 2.66 | 2.90 |

In this part of the experiment, nearly the same observations were acquired. The changes in percent weight loss, percent moisture and percent shrinkage were 0.16 %, 0.07 % and 0.10 %, respectively. The results revealed that experimental test conditions were uniform and did not change from one plate to another.

As relative humidity increased from 70 % to 75 %, percent weight loss decreased by 0.04 %, percent shrinkage did not deviate, nevertheless percent residual moisture increased by 0.04 % on the average.

**Table 4.16.** Percent average moisture, percent average shrinkage and percent average weight loss of all plates at different relative humidity levels

| % Relative Humidity | % Average Moisture | % Average Shrinkage | % Average Weight Loss |
|---|---|---|---|
| **60** | 0.55 | 2.95 | 17.95 |
| **65** | 0.61 | 2.94 | 17.89 |
| **70** | 0.64 | 2.93 | 17.86 |
| **75** | 0.68 | 2.93 | 17.82 |

The average percent weight loss varied from 17.82 to 17.95, the average moisture varied from 0.55 to 0.68 % and the average percent shrinkage varied from 2.93 to 2.95 for different percent relative humidity values as seen in Table 4.16.

The variation of percent average weight loss, percent average moisture and percent average shrinkage as a function of percent relative humidity for the plates dried at 100 $^{o}$C are given in Figure 4.9. The average value of percent moisture increased with increasing percent relative humidity. However, the average value of percent shrinkage and percent weight loss decreased when percent relative humidity increased to 75 % R.H. This behavior was interpreted as follows:

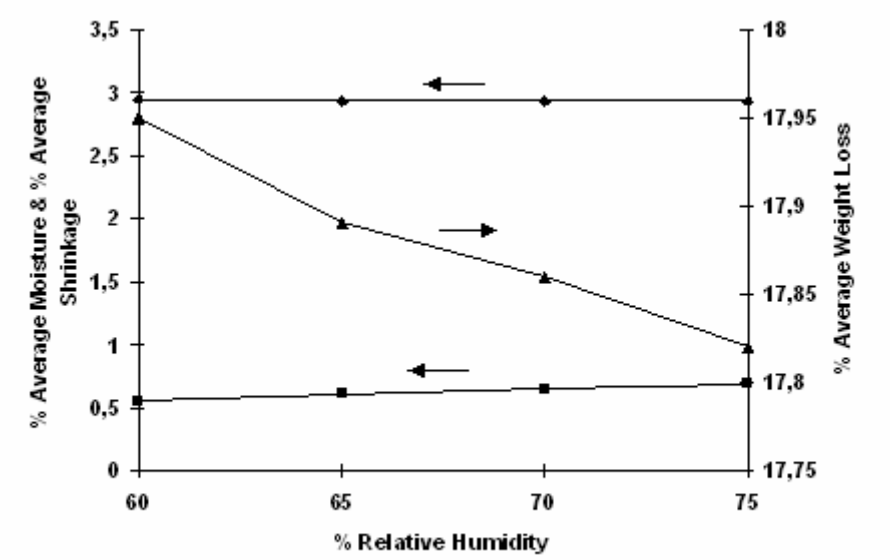



**Figure 4.9.** Variation of percent average weight loss, percent average moisture and percent average shrinkage with relative humidity for all plates

Dry air contains a low amount of water for a given temperature and has a low percent relative humidity. Wet air contains a high amount of water for a given temperature and has a high percent relative humidity. Dry air in contact with liquid water on a ceramic body surface will more aggressively encourage liquid to evaporate than will wet air. At a fixed temperature, a lower percent relative humidity encourages more evaporation than air at a higher relative humidity [3].

For many ceramic bodies, the first drying step is conditioning, which occurs in what is often called a 'conditioning chamber' or 'holding room'. This step although sometimes part of the manufacturing process, is not technically considered one of the drying stages. There is no water loss so no drying occurs. However, there are other subtle, but important phenomena at work during this process [21].

The conditioning process holds the ceramic body at a warm, ambient, constant temperature and at a high, constant humidity for a certain amount of time. This stage provides more time for water that was quickly added during the blending process to wet the body particles. Additionally, interior stresses generated during extrusion or handling that can later lead to cracking or warping can be relieved during this holding period. Conditioning also allows the internal temperature of the shape more time to warm up to be equal to the exterior temperature. This rises the temperature of all the water inside the shape. Since the viscosity of water significantly drops as the temperature increases, it becomes easier for the warmer water to migrate through the tight internal structure from the interior to the surface [21].

During the constant rate period, the shrinkage of the ware is equal to the volume of liquid evaporated [2]. The rate of drying depends on the water, which leaves the surface of the article and is absorbed and carried away by the air in the dryer. If this air is completely saturated (i.e. its humidity is 100 %) there will be no drying at all. If the air is stationary, drying will continue until the air is saturated and will then cease, only if the air is removed, both mechanically or naturally, will drying occur and the rate of drying will depend chiefly on that movement. The hotter the air, the more water will it hold and the more rapidly will it dry the wares, so long as it is not fully saturated, but air which is too hot and too dry may cause cracking [13].

By controlling the relative humidity in the air in contact with the surface of the ceramic body, the rate of evaporation, or water removal, from the ceramic body can be controlled. Controlling the air temperature and the relative humidity of the air are critical factors in controlling the drying process. The distribution and movement of that surrounding air is also an important factor. The volatized water film on the ware's surface must be removed to encourage further volatilization. Moving air across the wet surface encourages evaporation and scrubs the surface to remove the concentrated water molecules. With this reduced concentration of

the surface water, water from the interior of the ceramic piece is encouraged to migrate to the surface for removal by evaporation from the surface [3].

Based on the above results, it can be said that relative humidity in the beginning of the drying cycle should be less than 75 % because the moisture of the plates approaches to the critical value when the relative humidity becomes 75 %.

Spratford [20] investigated the effect of relative humidity on drying rate of slip cast pieces – large, hollow bowls that were 25 mm in diameter with an 8 mm wall thickness. The temperature in the plant ranged from 28 to 32 $^{o}$C, with humidity anywhere from 30 to 60 % R. H.

The values obtained for the effect of percent relative humidity on drying rate investigated in this study were comparable to those for the effect of percent relative humidity on drying rate of slip cast pieces – large, hollow bowls. He decreased the percent relative humidity by keeping the temperature of the dryer constant [20]. On reducing the percent relative humidity from 90 % to 70 %, it was seen that the weight loss increased and the elapsed time decreased from 144 h to 12 h. In the same manner, as percent relative humidity changed from 75 to 60 %, the average moisture changed from 0.68 to 0.55 % in this study.

## 4.7 BATCH COMPOSITION OF RAW MATERIALS

In this part of the work, the effect of non-plastics content in the slip recipe to the drying rate was studied. In order to see the effect of non-plastic usage in the slip recipe, the total amount of feldspar and quartz contents was changed in the prepared batch and two different slip recipes were prepared as discussed in section 3.1.2.

Plates were prepared and weighed. In order to speed up the drying for the plates, all plates were put to the laboratory dryer at 45 $^{o}$C for 20 h. They were weighed again. By measuring the dimensions of the plates, percent shrinkage values were calculated and the results are given in Table 4.17.

**Table 4.17.** Percent shrinkage measurements for Rp, $R_1$ and $R_2$ after taking the slip plates from the laboratory dryer at 45 $^{o}$C

| Plate Number | % Shrinkage | | |
|---|---|---|---|
| | $R_p$ | $R_1$ | $R_2$ |
| **1** | 2.88 | 2.91 | 2.77 |
| **2** | 2.87 | 2.92 | 2.80 |
| **3** | 2.88 | 2.92 | 2.80 |

As seen in Table 4.17, the percent shrinkage values for $R_p$ after taking the plates from the dryer was 2.88 with a deviation of ± 0.01%. Percent shrinkage for $R_1$ after taking the plates from the dryer was 2.92 with a deviation of ± 0.01 %. Percent shrinkage for $R_2$ after taking the plates from the dryer varied from 2.77 to 2.80 with a deviation of ± 0.03 %.

Afterwards, all plates prepared from $R_p$, $R_1$ and $R_2$ were dried at 100 $^oC$. They were weighed for every 30 minutes and the shrinkage calculations were carried out as seen in Tables 4.18 through 4.20.

**Table 4.18.** Percent shrinkage measurements for $R_p$ dried at 100 $^oC$

| Time (Min.) | % Shrinkage | | |
|---|---|---|---|
| | **Plate 1** | **Plate 2** | **Plate 3** |
| **0** | 2.88 | 2.87 | 2.88 |
| **30** | 2.88 | 2.88 | 2.90 |
| **60** | 2.90 | 2.92 | 2.93 |
| **90** | 2.95 | 2.96 | 2.97 |
| **120** | 3.00 | 3.00 | 3.00 |

For plate 1, percent shrinkage varied from 2.88 to 3.00, for plate 2 it varied from 2.87 to 3.00 and for plate 3, it varied from 2.88 to 3.00 as seen in Table 4.17. The average percent shrinkage was calculated as 3.00 after 2 h drying when $R_p$ was used.

**Table 4.19.** Percent shrinkage measurements for $R_1$ dried at 100 $^oC$

| Time (Min.) | % Shrinkage | | |
|---|---|---|---|
| | **Plate 1** | **Plate 2** | **Plate 3** |
| **0** | 2.91 | 2.92 | 2.91 |
| **30** | 2.93 | 2.95 | 2.94 |
| **60** | 2.97 | 2.95 | 2.98 |
| **90** | 3.00 | 3.08 | 3.04 |
| **120** | 3.12 | 3.14 | 3.09 |

As given in Table 4.19, for plate 1, percent shrinkage varied from 2.91 to 3.12, for plate 2, it varied from 2.92 to 3.14 and for plate 3, it varied from 2.91 to 3.09. The average percent shrinkage was calculated as 3.12 after 2 h drying when $R_1$ was used.

**Table 4.20.** Percent shrinkage measurements for $R_2$ dried at 100 $^oC$

| Time (Min.) | % Shrinkage | | |
|---|---|---|---|
| | **Plate 1** | **Plate 2** | **Plate 3** |
| **0** | 2.77 | 2.80 | 2.80 |
| **30** | 2.82 | 2.80 | 2.82 |
| **60** | 2.88 | 2.82 | 2.85 |
| **90** | 2.90 | 2.86 | 2.89 |
| **120** | 2.94 | 2.92 | 2.91 |

As given in Table 4.20, for plate 1, percent shrinkage varied from 2.77 to 2.94, for plate 2, it varied from 2.80 to 2.92 and for plate 3, it varied from 2.80 to 2.91. The average percent shrinkage was calculated as 2.92 after 2 h drying when $R_2$ was used.

After obtaining these results, moisture contents of the plates were calculated in accordance with the Equation 3.1 and shown in Tables 4.21 through 4.23.

**Table 4.21.** Results of percent weight loss, percent moisture and percent shrinkage for $R_1$

| Plate Number | % Weight Loss | | % Moisture | | % Shrinkage | |
|---|---|---|---|---|---|---|
| | Before placing into the dryer | After taking from the dryer | Before placing into the dryer | After taking from the dryer | Before placing into the dryer | After taking from the dryer |
| **1** | 10.80 | 17.71 | 7.94 | 1.03 | 2.91 | 3.12 |
| **2** | 10.21 | 17.64 | 8.53 | 1.10 | 2.92 | 3.14 |
| **3** | 10.02 | 17.59 | 8.72 | 1.15 | 2.91 | 3.09 |

When a comparison was made among the three plates, it was seen that there was slight deviation from one plate to another in testing parameters. That is, the changes in percent weight loss, percent moisture and percent shrinkage were 0.12 %, 0.12 % and 0.05 %, respectively.

From the point of view of testing parameters, it was observed that while percent weight loss increased, percent residual moisture decreased and percent shrinkage increased in the same manner for all plates as expected.

**Table 4.22.** Results of percent weight loss, percent moisture and percent shrinkage for $R_2$

| Plate Number | % Weight Loss | | % Moisture | | % Shrinkage | |
|---|---|---|---|---|---|---|
| | Before Placing into the Dryer | After Taking from the Dryer | Before Placing into the Dryer | After Taking from the Dryer | Before Placing into the Dryer | After Taking from the Dryer |
| **1** | 12.72 | 18.03 | 5.60 | 0.29 | 2.77 | 2.94 |
| **2** | 12.41 | 17.94 | 5.91 | 0.38 | 2.80 | 2.92 |
| **3** | 11.93 | 17.89 | 6.93 | 0.43 | 2.80 | 2.91 |

The changes in percent weight loss, percent moisture and percent shrinkage were 0.14 %, 0.14 % and 0.03 %, respectively for $R_2$, which means that the changes can be accepted as ignorable. As far as the testing parameters are concerned, the results revealed that as percent weight loss increased, percent residual moisture decreased and percent shrinkage increased in the same way for all plates as seen before.

When the results found for $R_2$ were compared to that of $R_1$, as non-plastic content increased by 10 %, percent weight loss increased by 0.10 %, nevertheless both percent residual moisture and percent shrinkage decreased by 0.20 % on the average.

**Table 4.23.** Results of percent weight loss, percent moisture and percent shrinkage for $R_p$

| Plate Number | % Weight Loss | | % Moisture | | % Shrinkage | |
|---|---|---|---|---|---|---|
| | Before placing into dryer | After taking from the dryer | Before placing into dryer | After taking from the dryer | Before placing into dryer | After taking from the dryer |
| **1** | 11.90 | 17.95 | 14.52 | 0.55 | 2.88 | 3.01 |
| **2** | 11.40 | 17.88 | 13.48 | 0.62 | 2.87 | 3.00 |
| **3** | 11.10 | 17.86 | 16.50 | 0.64 | 2.88 | 3.00 |

In a similar way, the results found for $R_p$ were more or less the same for the three plates. The changes in percent weight loss, percent moisture and percent shrinkage were 0.09 %, 0.07 % and 0.01 %, respectively. The results showed that as percent weight loss increased, percent residual moisture decreased and percent shrinkage increased in a similar way.

When the results found for $R_p$ were compared to that of $R_2$, a decrease of 5 % in non-plastic content gave rise to an increment of 0.05 % in percent weight loss, a decrease of 0.08 % in percent shrinkage and 0.23 % in percent residual moisture content on the average.

**Table 4.24.** Percent average weight loss, percent average moisture and percent average shrinkage change of all plates for different slip recipes

| Recipe | % Non-plastic Content | % Average Moisture | % Average Shrinkage | % Weight Loss |
|---|---|---|---|---|
| $R_p$ | 50 | 0.60 | 3.00 | 17.90 |
| $R_1$ | 45 | 1.09 | 3.12 | 17.65 |
| $R_2$ | 55 | 0.37 | 2.92 | 17.95 |

As given in Table 4.24, for $R_1$, $R_2$ and $R_p$, the percent average moisture content changed from 0.37 to 1.09 %, the percent average shrinkage varied from 2.92 to 3.12 % and the percent weight loss changed from 17.65 to 17.95.

The variation of percent average moisture content, percent average weight loss and percent average shrinkage as a function of non plastic content for the plates prepared by $R_p$, $R_1$ and $R_2$ are shown in Figure 4.10. The average value of percent moisture and percent shrinkage decreased with increasing non-plastic content. On the other hand, the average value of percent weight loss increased with increasing non-plastic content. This behavior was interpreted as follows:

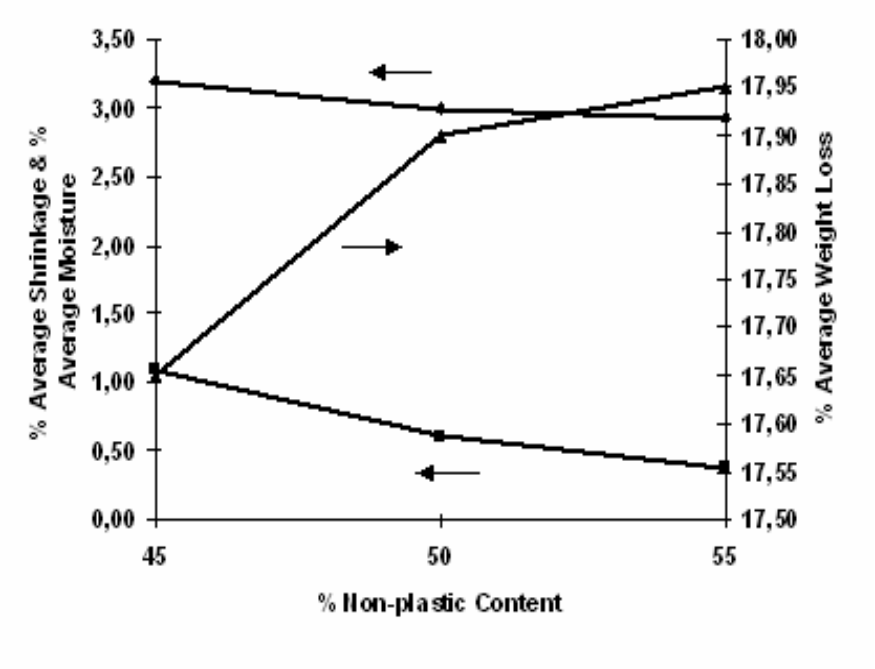



**Figure 4.10.** The effect of non-plastics on percent average moisture, percent average shrinkage and percent average weight loss

As soon as the body particles are extracted from the mould, they are surrounded by a capillary lattice of water that during drying has to migrate to the surface of the piece in order to evaporate, without damaging the integrity of the semi – processed product. Therefore, the two fundamental factors controlling drying are the water diffusion speed through the thickness of the piece and the evaporation speed. The diffusion speed of water into the capillary canals depends on body composition. The richer in plastic and very fine materials, the slower the diffusion. A more plastic body is also prone to greater shrinkage and therefore the water passage canals are obstructed to a greater extent with a consequent reduction in diffusion speed [6].

The selection of the type and quantity of clay and kaolin must be made not only in consideration of the thickness formation time, but also by evaluating how it conditions the following characteristics of the body [22]:

1) Unfired shrinkage and drying speed. Clays with a greater plasticity increase shrinkage and drying difficulties.
2) Fired shrinkage and deformation and vitrification rate of the body.

Using a higher proportion of non – plastic large grained material in the raw material batch may reduce the shrinkage. Such additions have a dual beneficial action; they reduce the overall content of colloidal material and they also build up a rigid skeleton throughout the mass, which reduces shrinkage and preserves an open structure, which facilitates drying. Sand and grog are frequently employed for this purpose [8].

As shrinkage is equal in volume to that of water removed during the constant rate period, i.e., most of it, it is clear that the more water in the body, the more occasion there is for drying cracks. The water is chiefly bound up with plastic clays and hence they must be used discriminately, replacing them with non – plastic raw materials [11].

Thin ceramic products are molded from mixtures consisting of argillaceous matter and non – plastic materials. The particle size of the clays and kaolins usually lies within the limits of colloidal dimensions or more, and the particles of non – plastics are considerably larger. The more non – plastic material there is in the mixture and the more coarsely it is ground, the greater will be the cross section of the capillaries in the mixture and the higher the rate of transfer of the moisture to the surface, and consequently the more rapidly the ware can be dried without the cracks and deformation. Shrinkage of the ware ceases as soon as the evaporation of the so-called shrinkage moisture is complete [10].

Excessive shrinkage is undesirable as it tends to cause cracking and distortion of the ware. The commonest cure is to add non-plastics to the clay. These relatively coarse materials simply reduce the number of water films per unit distance by displacing a group of clay particles and their associated films by an equal volume

of stable particles. In the same way, coarse-grained clays shrink less than fine-grained ones. Because of the orientation of the clay plates, cast bodies shrink less than plastic ones [23].

Clays are composed of inherently fine – grained particles which would be expected to have a fine capillary pore system, hence the amount of retained water will be greater than in sand where the particles are larger and more rounded in shape. The shrinkage in clay bodies at water contents below the critical point is, however, relatively small compared with the amount in the constant period of drying [13].

Taçyıldız [24] explained the effects of materials composition on the physical properties of sanitary ware samples. The experiments were carried in two groups; firstly, the mineralogical composition of feldspar in the mixture was taken constant and the effects of silicate and kaolin on the physical properties of sanitary ware were investigated. In the second group of experiments, the mineralogical composition of feldspar in the mixture was increased while the ratio of silicate to kaolin was kept constant.

The values obtained for the effect of non – plastic content on drying rate investigated in this study were comparable to those for the effect of non – plastic content on drying rate of sanitary ware samples. Taçyıldız [24] increased the content of non – plastics by increasing feldspar and quartz ratios. As the feldspar content increased from 13 to 50 %, the percent shrinkage changed from 3.20 to 1.76 and as the quartz to kaolinite ratio changed from 0.56 to 3.5, percent shrinkage decreased from 3.20 to 0.01 %. In the same manner, as the non – plastic content increased from 45 % to 55 %, the percent shrinkage decreased from 3.12 to 2.92 in this study.

# CHAPTER 5

# CONCLUSIONS

1. The residence time for the green ware before placing into the dryer was effective on drying rate. The optimum residence time for the greenline at 30 $^{o}$C and 60 % relative humidity was found as 6 h. As the residence time increased, percent weight loss and percent shrinkage increased.

2. The optimum temperature in the dryer was 100 $^{o}$C. Lower temperatures, i.e. 80 and 90 $^{o}$C were not sufficient to obtain the desired value of percent moisture level for the wares at a given time period.

3. To obtain an optimum moisture level, 8 h drying cycle at 100 $^{o}$C was sufficient.

4. Relative humidity in the dryer should be less than 75 %. Otherwise, the residual moisture level within the ware would be too high for 12 h tunnel kiln firing.

5. As the non-plastic content in the slip recipe increases, residual moisture content and percent shrinkage decrease in view of reducing the number of water films per unit distance by displacing a group of clay particles and their associated films by an equal volume of stable particles.

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# APPENDIX A

**Table A.1.** Percent weight loss of the plates prepared from the recipe Rp after 24-hour residence time prior to placing them into the dryer

| Residence Time (h) | % Weight Loss | | |
|---|---|---|---|
| | **Plate 1** | **Plate 2** | **Plate 3** |
| **0** | 0.0 | 0.0 | 0.0 |
| **1** | 1.2 | 1.3 | 1.1 |
| **2** | 2.9 | 2.9 | 2.8 |
| **3** | 3.4 | 3.5 | 3.5 |
| **4** | 4.7 | 4.8 | 4.7 |
| **5** | 5.9 | 5.8 | 5.9 |
| **6** | 6.6 | 6.5 | 6.5 |
| **7** | 6.9 | 7.0 | 7.1 |
| **8** | 8.2 | 8.1 | 8.4 |
| **9** | 9.2 | 9.0 | 9.3 |
| **10** | 9.5 | 9.5 | 9.7 |
| **11** | 10.2 | 10.4 | 10.3 |
| **12** | 11.1 | 11.0 | 11.2 |
| **13** | 11.9 | 12.0 | 12.4 |
| **14** | 13.3 | 13.1 | 13.2 |
| **15** | 13.5 | 13.7 | 13.8 |
| **16** | 14.0 | 14.2 | 14.1 |
| **17** | 14.8 | 14.6 | 15.0 |
| **18** | 14.9 | 15.0 | 15.1 |
| **19** | 15.4 | 15.3 | 15.5 |
| **20** | 15.7 | 15.6 | 15.8 |
| **21** | 15.9 | 16.0 | 16.0 |
| **22** | 16.1 | 16.2 | 16.3 |
| **23** | 16.3 | 16.5 | 16.6 |
| **24** | 16.6 | 16.7 | 16.8 |

**Table A.2.** Percent moisture level of the plates prepared from the recipe Rp after 24-hour residence time prior to placing them into the dryer

| Residence Time (h) | % Moisture Level | | |
|---|---|---|---|
| | **Plate 1** | **Plate 2** | **Plate 3** |
| **0** | 18.5 | 18.5 | 18.5 |
| **1** | 17.3 | 17.2 | 17.4 |
| **2** | 15.6 | 15.6 | 15.7 |
| **3** | 15.1 | 15.0 | 15.0 |
| **4** | 13.8 | 13.7 | 13.8 |
| **5** | 12.6 | 12.7 | 12.6 |
| **6** | 11.9 | 12.0 | 12.0 |
| **7** | 11.6 | 11.5 | 11.4 |
| **8** | 10.3 | 10.4 | 10.1 |
| **9** | 9.3 | 9.5 | 9.2 |
| **10** | 9.0 | 9.0 | 8.8 |
| **11** | 8.3 | 8.1 | 8.2 |
| **12** | 7.4 | 7.5 | 7.3 |
| **13** | 6.6 | 6.5 | 6.1 |
| **14** | 5.2 | 5.4 | 5.3 |
| **15** | 5.0 | 4.8 | 4.7 |
| **16** | 4.5 | 4.3 | 4.4 |
| **17** | 3.7 | 3.9 | 3.5 |
| **18** | 3.6 | 3.5 | 3.4 |
| **19** | 3.1 | 3.2 | 3.0 |
| **20** | 2.8 | 2.9 | 2.7 |
| **21** | 2.6 | 2.5 | 2.5 |
| **22** | 2.4 | 2.3 | 2.2 |
| **23** | 2.2 | 2.0 | 1.9 |
| **24** | 1.9 | 1.8 | 1.7 |

**Table A.3.** Percent shrinkage of the plates prepared from the recipe Rp after 24-hour residence time prior to placing them into the dryer

| Residence Time (h) | % Shrinkage | | |
|---|---|---|---|
| | **Plate 1** | **Plate 2** | **Plate 3** |
| **0** | 0.0 | 0.0 | 0.0 |
| **1** | 1.0 | 1.1 | 1.0 |
| **2** | 1.5 | 1.4 | 1.5 |
| **3** | 1.9 | 2.0 | 1.8 |
| **4** | 2.3 | 2.2 | 2.4 |
| **5** | 2.8 | 2.7 | 2.7 |
| **6** | 2.9 | 2.9 | 3.0 |
| **7** | 3.0 | 3.0 | 3.0 |
| **8** | 3.0 | 3.0 | 3.0 |
| **9** | 3.0 | 3.0 | 3.0 |
| **10** | 3.0 | 3.0 | 3.0 |
| **11** | 3.0 | 3.0 | 3.0 |
| **12** | 3.0 | 3.0 | 3.0 |
| **13** | 3.0 | 3.0 | 3.0 |
| **14** | 3.0 | 3.0 | 3.0 |
| **15** | 3.0 | 3.0 | 3.0 |
| **16** | 3.0 | 3.0 | 3.0 |
| **17** | 3.0 | 3.0 | 3.0 |
| **18** | 3.0 | 3.0 | 3.0 |
| **19** | 3.0 | 3.0 | 3.0 |
| **20** | 3.0 | 3.0 | 3.0 |
| **21** | 3.0 | 3.0 | 3.0 |
| **22** | 3.0 | 3.0 | 3.0 |
| **23** | 3.0 | 3.0 | 3.0 |
| **24** | 3.0 | 3.0 | 3.0 |