EFFECT OF BINDERS AND PLASTICISERS ON ALUMINA PROCESSING

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PHILOMINA EKKA
ROLL 108CR021

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CONTENTS

CERTIFICATE

This is certified that the work contained in the project entitled “EFFECT OF BINDERS AND PLASTICISERS ON ALUMINA PROCESSING” by Philomina Ekka (Roll no. 108CR021) in partial fulfillment of the requirements of the award of Bachelor of Technology Degree in Ceramic Engineering at the National Institute of Technology, Rourkela is an authentic work carried out by her under my supervision and guidance. To the best of my knowledge, the matter embodied in the thesis has not been submitted to any other university / institute for the award of any Degree or Diploma.

S.BHATTACHARYYA
PROFESSOR
Department of Ceramic Engineering
National Institute of Technology
Rourkela-769008

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Philomina Ekka
Roll No. 108CR021

ABSTRACT

During ceramic processing several different additives must be introduced into the batch for producing particle dispersion and flow behaviour necessary for forming. These additives unlike the powder or the solvent are added in a very minor quantity, most of which is eliminated in the subsequent processing steps. In this project, different combinations of plasticiser (Polyethylene glycol) and binder (Polyvinyl alcohol) have been used for forming Alumina ceramics. PVA and PEG have been one of the most conventional pairs of binder and plasticiser used on Alumina system. This project aims to study the variation of these properties with the plasticiser amount. It was observed that with the increase in the plasticiser amount, the green density increased till it reached a maximum and then became independent of further additions, in some cases, even decreasing. This high green density led to high sintered density too. But the compositions having more than 0.5% PEG in spite of high green density, showed poor sintered density indicating that, as PEG vaporised out, it left pores, and even some residues which hindered densification while sintering. Therefore, the final density of the product depended on two mutually opposite factors: the high green density and the high amount of impurity left after PEG vaporised. Fired strength (like fired density) was the best for unplasticised sample. Still, highest green strength corresponded to 0.25% and 0.5% plasticiser (PEG) necessitating its use in green products. Green density showed an increasing trend, which reversed after firing, limiting the use of PEG to 0.5% in the batch.

LIST OF FIGURES

LIST OF TABLES

CHAPTER 1. INTRODUCTION

INTRODUCTION

In ceramic processing, additives used to bridge the particles together using any mechanism is classified as a binder. These additives may also serve other purposes too. The paste prepared is called workable when it is just suitable for molding, shaping and further processing steps. This workable system is often produced on using only one particular flocculating additive, but in some cases two or more different types are used.

1.1 Different types of binders

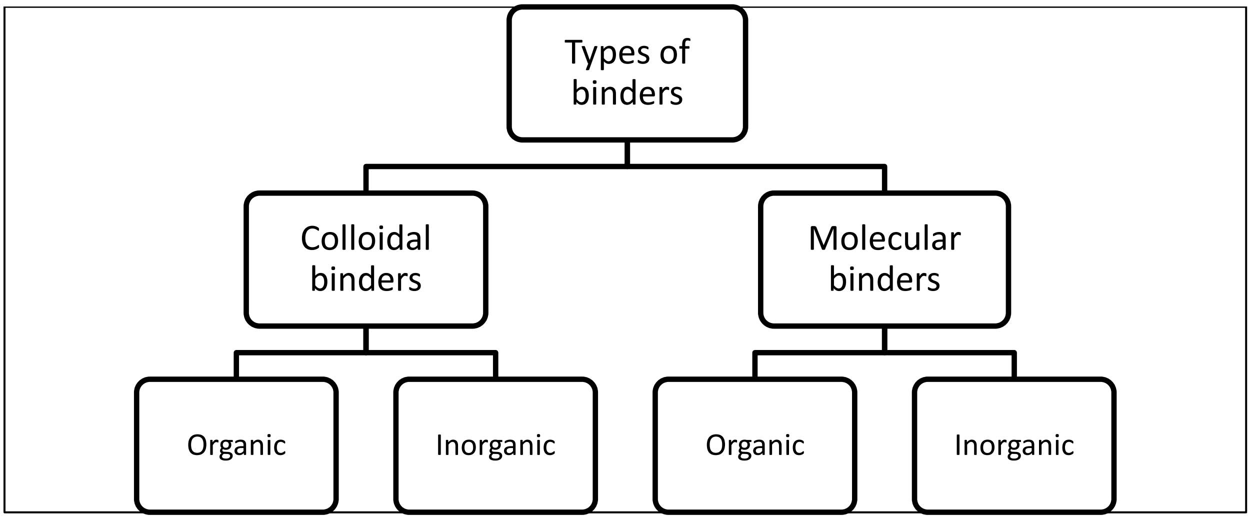

Fig.1.1 Types of binders[1]

1.1.1 COLLOIDAL BINDERS

Clay minerals were used as colloidal binders in processing ancient ceramics, at a time when even the concept of binders did not exist. Today, they are also used in advanced ceramics, where Alumina and Silica are acceptable in the composition. Example of clay binders are fine Kaolin, Ball clay, and Bentonite. These binders now come more refined to produce consistent behaviour[1].

Microcrystalline Cellulose is an organic colloidal particle binder. It is manufactured from high-purity Cellulose pulp and may be used when submicron pores are desired or are not a problem[1].

1.1.2 MOLECULAR BINDERS

Molecular binders range widely in composition and may be natural or synthetic substances, thus offering a lot of options for modifying the rheological behaviour of the paste.

When decomposed under oxidising conditions, synthetic organic binders introduce relatively little inorganic impurity unlike inorganic binders which leave behind a lot of residue. Hence these inorganic binders can be used only in systems where the (inorganic) residue component is compatible with the particle composition[1].

Some systems can also use two types of binders, i.e. Molecular binders with refined Clay ones[1]. Inherently Ball clay contains both clay minerals and natural organic matter of both a colloidal and molecular nature which influences its properties. Two molecular binders i.e. PVA and Starch have been used in our experiments.

1.2 POLYVINYL ALCOHOL

Polyvinyl alcohol or PVA (PVOH) is a very popular adhesion binder, which acts by getting adsorbed on the particles[1]. This polymer has a basic repeating structural unit called the mer. The no. of mers in the molecule is indicated by the subscript n which is the degree of polymerization. The molecular weight of a polymer increases as the degree of polymerization increases.

Amorphous density at 25^{o}C: 1.26 g/cm^3[2]

Crystalline density at 25^{o}C: 1.35 g/cm^3[2]

Molecular weight of repeating unit: 44.00 g/mol

1.2.1 Molecular structure

In the PVA structure, the –C–C– linkage is referred to as the vinyl backbone, and the –H and –OH are referred to as the side groups. Vinyl binders with the –C–C– backbone are very flexible binders. The polar –OH group is hydrophilic, which promotes initial wetting and dissolving of the PVA in a polar liquid such as water. Hydrogen bonding of the –OH side group to the surface of a particle provides adhesion, and the dipolar attraction of –OH side groups produces intermolecular bonding. PVA is a binder which has a strong affinity for adsorption on oxide particles dispersed in water[1].

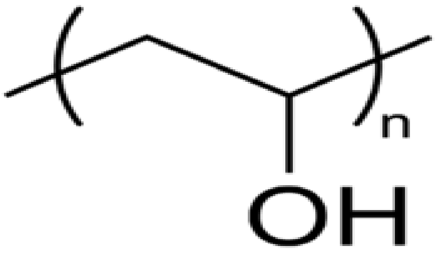

Fig. 1.2. Molecular structure of fully hydrolyzed PVA[2].

1.2.2 Mechanism

Molecular binders are low- to high- molecular weight polymer molecules which may adsorb on the surfaces of particles and bridge them together, like PVA which has hydrogen bonds or form a polymer-polymer bonded network (film) among the particles, i.e. waxes. Accordingly there can be two types of molecular binders: adhesion and film binders. They can be non-ionic, anionic, or cationic. PVA is a non-ionic (adhesion) binder[1].

The physical properties of PVA, such as the strength, water solubility, gas permeability and thermal characteristics vary with the degree of crystallinity, which is in turn heavily dependent on the degree of hydrolysis and the average molecular weight of the polymer. The residual acetate groups in partially hydrolyzed PVA reduce the overall degree of crystallinity. This results in materials with lower strength and increased water solubility than the fully hydrolysed grades. The partially hydrolysed grades may be considered as copolymers, while those that are fully hydrolysed may be considered as homopolymers. In our project and generally for all laboratory purposes, fully hydrolysed grade is used[3].

1.2.3 Other binders

Polyethylene (PE), Polypropylene (PP), and Polystyrene (PS), used in injection moulding systems are polymer resins. More commonly known as plastics, they are usually added to form a bonding matrix, which requires that they must be mouldable at an early stage and later capable of being hardened to form a structural bond[1].

Other types of additives that can be used like binders are reaction bonding cements and hydraulic cements[1].

1.3. PLASTICISERS

A plasticiser is added to modify the viscoelastic properties of a condensed binder-phase film on the particles. The moldability of a binder is very dependent on temperature. At about 20°C PVA is elastic and brittle and two separate films bond very poorly when pressed together; movement of the molecules is very limited, and the brittle binder is said to be in a glassy state. At about 90°C, the thermal energy is sufficient to enable some of the molecules to flow and realign when compressed, and bonding between films occurs. This is called the rubbery state. The temperature between these two states at which the deformation changes from elastic behavior (fracture at a strain greater than about 5%) to time–dependent viscoelastic deformation(no fracture at a strain exceeding 100%) is called the Glass Transition Temperature(T_g) and is shown in Fig.3. Polymer films exhibit a change in resistance to mechanical deformation at the glass transition temperature which can be used to determine the same. On heating to a temperature above the rubbery state, molding produces viscous flow behavior. A plasticiser helps in bringing down the glass transition temperature. PVA generally has a T_g of 85°C[2]. Even minor amounts of PEG can drastically bring down this temperature[1].

The most beneficial formulations producing highly fluid behavior are those with a low yield stress, a low consistency coefficient, and a high flow behavior index.

$$\tau = \tau_o + K(\Upsilon)^n \ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots\ldots 1.1$$

Where τ is the shear stress, τ_o is the yield stress, K is the consistency coefficient, Υ is the strain rate, and n is the flow behavior index. This model is convenient for analysis of rheological behavior over a wide range of fluids that are either Newtonian (n=1), shear-thinning ($0 < n < 1$), or shear-thickening ($1 < n$)[1].

A good flow ensures good mass transfer thus leading to easy rearrangement of molecules.

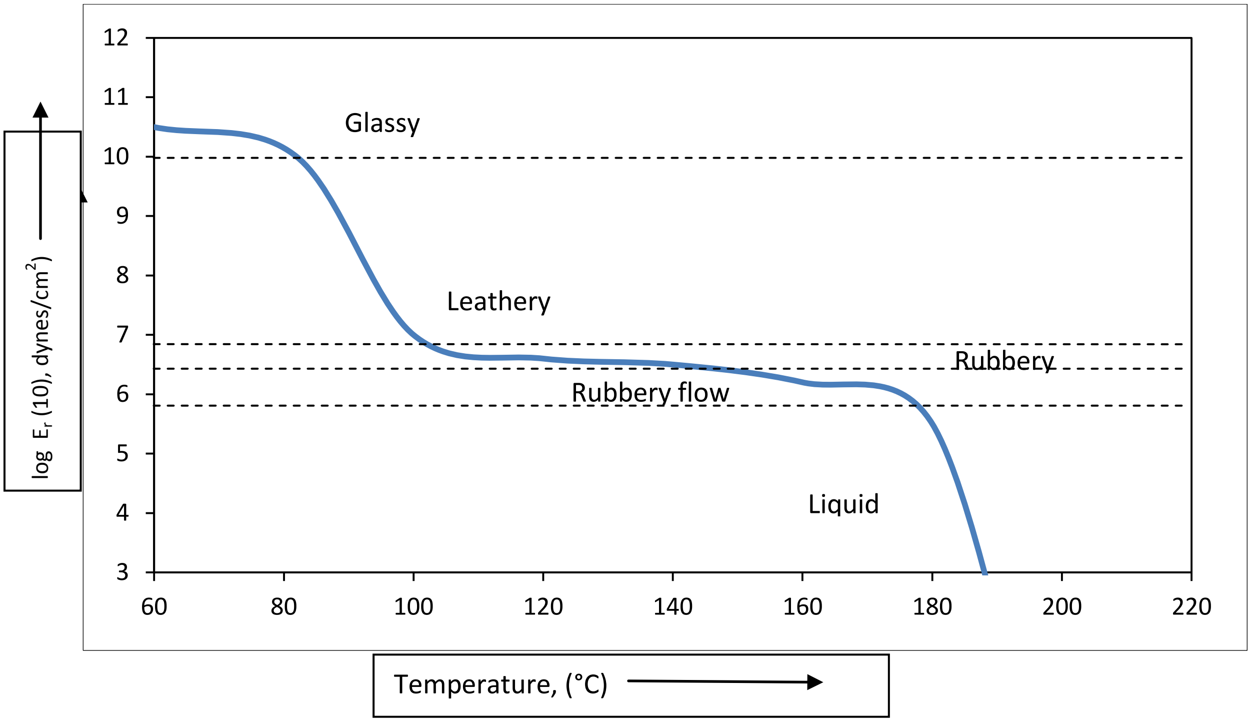

Fig. 1.3. Variation of relaxed elastic modulus of Polystyrene with increased temperature and regions of different viscoelastic behavior[1].

1.4. POLYETHYLENE GLYCOL

Polyethylene glycol is a commonly used plasticiser. It is also used as an additive to adhesives or printing inks. It is soluble in various organic solvents, e.g. in alcohol, ether and benzene[1].

1.4.1 Molecular structure

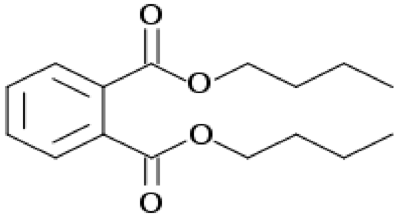

Fig.1.4. Structure of Polyethylene glycol[2]

1.4.2 Mechanism

Ceramic systems containing a binder are commonly molded above the glass transition temperature of the binder. Small molecules distributed among the larger polymer molecules cause the polymers to pack less densely and reduce the Vanderwaal’s forces binding the polymer molecules together. The presence of the small “plasticising” molecules softens and increases the flexibility of the binder but also reduces its strength. The plasticiser thus effectively reduces the T_g of the polymer[1].

1.4.2 Other additives used as Plasticisers

Water is also used as a plasticiser. It can be used combined with other plasticisers too. In case of PVA binder systems, water is always used to incorporate PVA in the system, thus automatically plasticising the mix.

CHAPTER 2. LITERATURE REVIEW

LITERATURE REVIEW

Romdhane et al.[4] explained in his paper that the combined interaction between a dispersant and a binder is very complex. And often depends on the competitive tendency between these molecules to get absorbed on the Alumina particles. In order to avoid or minimize this problem copolymers can be used, whose ionic part serves as a dispersant and neutral part as a binder. A new copolymer, PVX was designed which contained one carboxylate part (-COO-) and one hydroxyl part (-OH-) to serve the double function of a dispersant and a binder. Different PVX molecules with different percentages of the carboxylate group were tried, and PVX-35 with 35% Carboxylate group were found to give the best results as compared to PVA and even plasticised PVA (PEG added). PVX-35's glass transition temp. (T_g) was determined to be 35° C which is much lower than the T_g of dry PVA. In spray-drying, PVA migrates and segregates on the surface of the Alumina particles as a hard polymeric shell while PVX-35 formed a ductile shell which had better inter-diffusion while forming. Also, the PVX-35 adsorption percentage on Alumina particles was much better than for the classical PVA-PEG mix.

S. Baklauti et al.[5] reported in his paper that the amount and the nature of the binder have a huge effect on the Young's modulus of the green compact. And thus on the mechanical strength of the compact. They found out that if the binder is removed from the pressed sample by thermal treatment, the young's modulus decreased down to a very low value, rendering the fired product brittle. To avoid this, some lignin derivatives can be added to binders like Polyvinyl alcohol which increase the activation energy of the thermal degradation reactions of the binder, thus protecting it, as suggested by D.M. Fernandes et al[6]. The available literature for calculating the Young's modulus for two-phase materials is not applicable here because the pressed samples consist of three phases: Alumina, the binder and the porosity[5]. In our case, a fourth phase was the plasticiser, PEG.

Compacts of high DPO (Degree of Particle Orientation), i.e. higher number of particles aligned in the same direction, are expected to show higher shrinkage in the direction normal to the alignment. This mostly happens when the particles are elongated and uniaxial pressing is used to compact them. These particles during pressing tend to have a particular orientation to help compaction. Therefore, a significant anisotropic shrinkage may also be expected during sintering for elongated particles[7]. The reactive Alumina that we used was mostly spherical in shape.

There have been some apprehensions about organic plasticisers too. There are serious safety and environmental concerns with the evaporation and debinding of these organics, which traditionally include alcohols, ketones, polyethylene wax, vinyl additives, and others. Marek Sikora et al.[8] reported that the diffusion of these organic binders and plasticisers during heating and the gases produced during pyrolysis can cause unwanted cracks and shape distortion in pre-sintered parts. Relatively long heat treatments (e.g. up to one week at 200°C and above) are typically needed to complete pyrolysis. Higher rates of heating produce internal stresses that commonly cause cracks and shape distortion. Another concern with traditional, organic additives is that not all of the decomposition products of pyrolysis are removed by evaporation and gaseous diffusion; it is possible that incomplete combustion can leave behind carbonaceous residues which will contaminate microstructures. Some researches today emphasise the need for finding out water soluble additives that replace these organic chemicals and evaporate safely without causing cracks, shape distortion, or microstructure contamination in sintered parts while acting like a good plasticiser too.

Samir Baklauti et al.[9] reported in another paper that the small fraction of the acetate side groups (~10 mol %) actually stabilise the polymer PVA by preventing the depolymerisation reaction[9]. So, partially hydrolysed PVA is a better option than fully hydrolyzed PVA. In his paper he studied the affect of binder removal on the mechanical strength of alumina green samples.

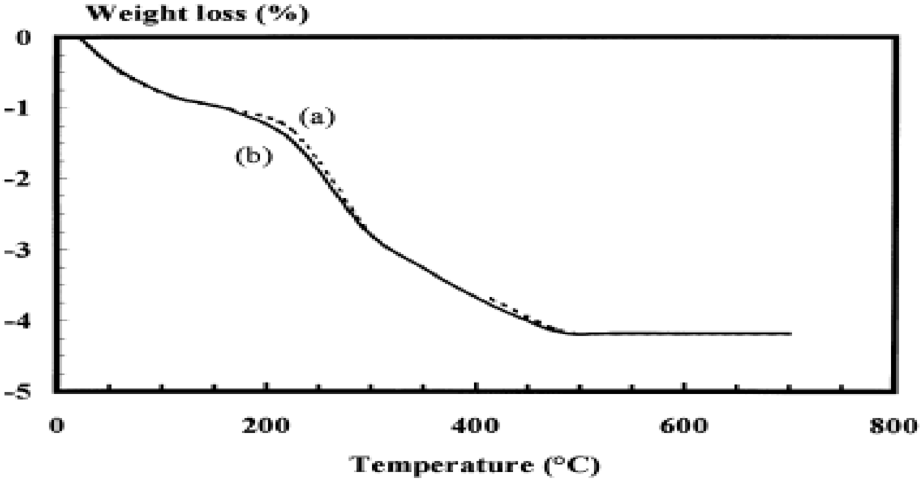

Fig.2.1 TG plot of (a) the spray-dried Alumina powder containing 3 wt. % PVA and (b) of a sample prepared from the same powder by uniaxial pressing at 70 MPa with a relative density about 57%[9].

Mechanical strength was tested on the as-received Alumina compact and spray dried Alumina compact (spray dried with PVA). The spray dried Alumina compact was more sensitive to the temperature change. The compact, as expected, showed higher green strength and increased from 20°C to 200°C, which in the TG plot, Fig.5, marks the elimination of water. Water which was plasticising the mix, also weakened the green compact. So when it left, there was an increase in the strength of the powder. Presence of acetic groups on the partially hydrolyzed PVA protected it from depolymeristaion to some temperature. There was a maximum and then there was a decrease in the strength due to the elimination of water and acetic acid from the polymer and the oxidation of the vinyl chains. This continued till both the strengths came down to the same level, where sintering started. For the spray dried Alumina, this indicates that now two alumina particles are in contact with each other and there’s no binder film in between. When sintering starts, the as-received Alumina sinters at a faster rate, may be because of the absence of the carbonaceous residue of the polymer and difference in the microstructure. Even at high temperature, this residue existed and reduced the strength of the spray dried Alumina as shown in Fig. 6.

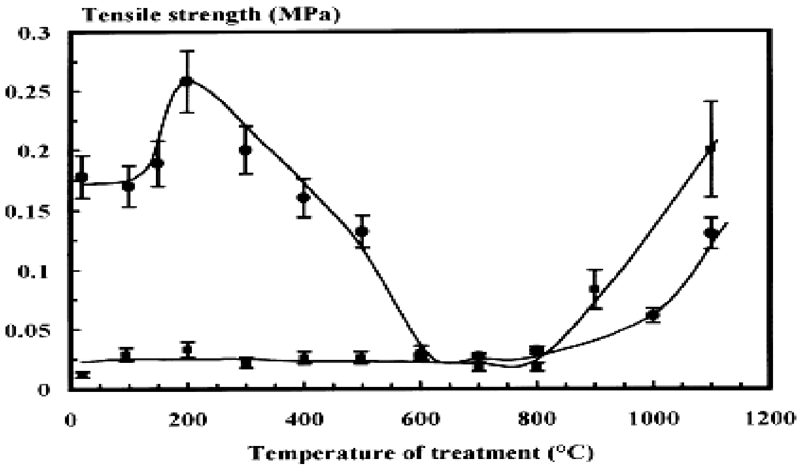

Fig.2.2. Evolution of the strength of the compacts with a relative density about 57% prepared from (●) as received and (■) from the spray dried Alumina versus temperature of treatment[9].

CHAPTER 3. EXPERIMENTAL WORK

EXPERIMENTAL WORK

3.1 Batch formulation

BATCH CALCULATION FOR MOTHER BATCH (MAY)

TABLE 3.1. Batch requirement

No. of pellets	Property/Characteristics
3	Green CCS, density analysis
6	Fired diametral compressive strength, fired density analysis
1	SEM analysis
6	Extra

Total no. of pellets = (16 pellets) * (7 compositions: 0, 0.25, 0.5, 0.75, 1, 1.25, and 1.5)

= 112 pellets

We have a 12.5 mm diameter die, radius = 6.25 mm

Height of the pellets, as required: 6 mm

Volume of the pellets = 3.141 * $(\text{Radius})^2$ * (Height)

= 3.141 * 6.25^2 * 0.6 cc

Theoretical density of Alumina = 3.98 gm/cc

Theoretical mass required = Density * Volume

Due to porosity, experimental density achieved is always lesser than the theoretical density.

Therefore, experimental mass of 1 pellet = (Theoretical mass)/2, from (2), (3) and (4)

= 1.4653 gms

Therefore, the total mass required = 1.4653 each pellet * 112 pellets

= 164.1136 gms.

Taking into account all the procedural mass loss, we made a batch of 180 gms.

BATCH CALCULATION FOR PVA AND PEG

For making 8% and 4% PVA and PEG respectively, 8 and 4 gms of each powder were taken in beakers containing 100 ml of distilled water. Following is the preparation technique.

There were 25 gms of batch powder for each composition. So, 4% PEG solution was added according to the following calculation:

For 0.25% PEG composition, 100 gms of Alumina require 0.25 gms of PEG

Or, 1 gm requires 0.25/100 gms = 0.0025 gms of PEG

Therefore, 25 gms require 0.0025*25 gms = 0.0625 gms

In a 4% PEG solution, 1 gm PEG is in 25 ml of solution.

So, 0.0625 gm PEG is in 25* 0.0625 ml = 1.5625 or 1.6 ml of solution.

PEG mass required for 25 gms

TABLE 3.2. Batch calculation for PEG

Composition	PEG mass required for 25 gms Alumina (in gms)	Volume of PEG solution required (in ml)
0.25%	0.0625	1.6
0.5%	0.125	3.1
0.75%	0.1875	4.7
1%	0.25	6.3
1.25%	0.3125	7.8
1.5%	0.375	9.4

3.2 Sample preparation

3.2.1 Preparation of PVA solution

The following are the steps taken to make PVA:

- According to the batch calculation, required amount of distilled water was taken in a beaker.
- It was heated on a magnetic stirrer, simultaneously stirred.
- The weighed amount of PVA was added in small parts, added more only when the previous addition had dissolved.
- It was ensured that the temperature remained below 85°C. Usually, constant heating is not required and it was heated in intervals.
- Sometimes extra water was also added to keep up the required percentage of water.
- Continuous stirring resulted in a clear transparent solution which was cooled and stored in an airtight bottle.
- If in case the solution turns yellow, it is an indication that the PVA has burnt and needs to be prepared again.

3.2.2 Preparation of PEG solution

The following are the steps taken to make PEG:

- According to the batch calculation, required amount of distilled water was taken in a beaker.
- The weighed amount of PEG was added into the beaker.
- The solution was continuously stirred till the entire PEG dissolved.
- The solution was stored in an airtight container.

3.2.3 Preparation of Stearic acid

The following are the steps taken to make stearic acid:

- According to the batch calculation, required amount of Isopropyl alcohol was taken in a beaker.
- The weighed amount of Stearic acid was added into the beaker.
- The solution was continuously stirred till all of the stearic acid dissolved.
- The solution was stored in an airtight container.

3.2.4 Pellet making

The following are the steps taken to make Alumina batch:

- The required amount of reactive Alumina powder of 0.75μ size was taken in an agate mortar.
- With constant stirring of the pestle, the prepared PVA was added into the mortar. All the agglomerates were broken and the mixture was made homogeneous with stirring.
- The viscous paste was dried in a vacuum oven. The agate mortar should be covered but it should allow enough passage of air to let out water vapour. The temperature should not exceed 70-80°C. Prolonged drying at high temperatures (>100°C) may cause the PVA to char.
- After it dried completely, it was scraped out and weighed to get divided into 7 parts symbolising 7 different compositions.
- The PEG solution was added into these compositions according to the batch calculations.
- The 6 batches were allowed to dry at a temperature below 80°C (1 batch contained no PEG).
- From each composition, powder mass for 11 pellets was separated out.

3.2.4 Pressing

The following are the steps taken to make Alumina pellets:

- The Hydraulic Carver Press was setup for a pump speed of 25%, load of 2.5 Ton and a dwell time of 120 seconds.
- A high carbon, high chrome steel die of 12.5 mm diameter was used.
- Acetone was used to clean the die and 4% stearic acid was used for lubrication.
- Altogether 77 pellets were pressed (7 X 11).
- The pellets were dried in a vacuum oven at 70°C for 2 hours.

3.2.4 Sintering of the pellets

The pellets were sintered through the following steps:

- 48 pellets were sent for firing.
- The pellets were fired at 500°C for 1 hour and at 1600°C for 2 hours (Fig. 3.1).
- The furnace used was Raising Hearth furnace.
- The base plate used in this process was made of Alumina.

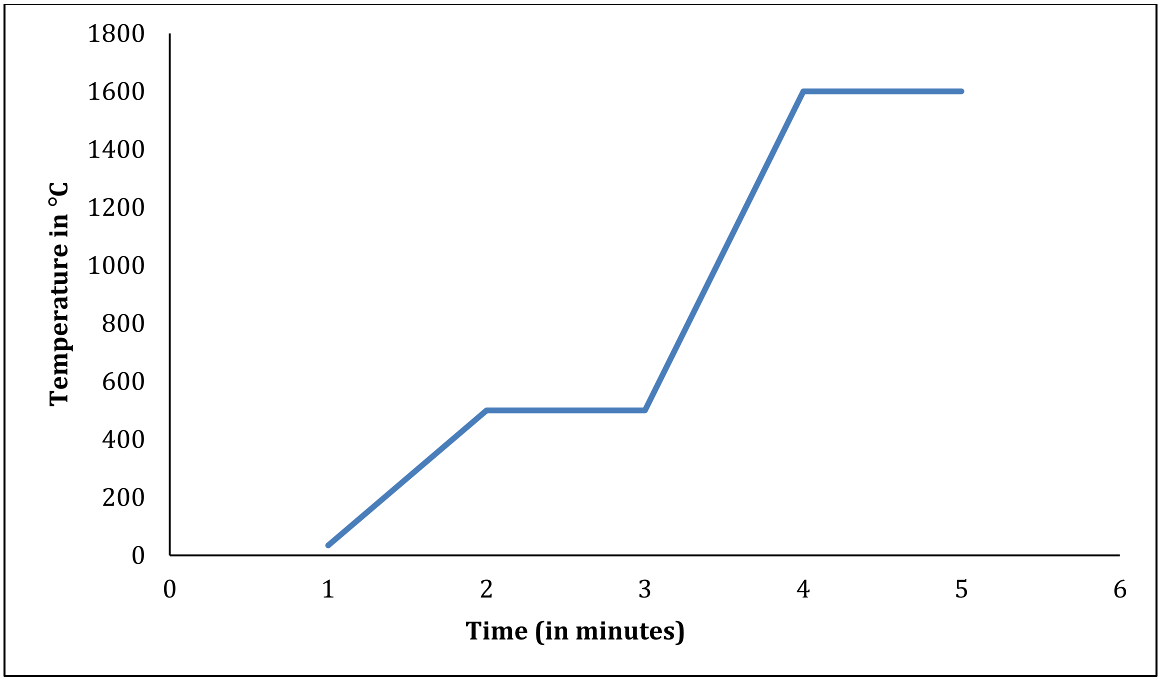

Fig.3.1. The firing schedule: time vs. temperature.

3.2.5 Optimization of dwell pressure

The following are the steps for optimising the dwell pressure:

- The optimum dwell pressure range is 120 to 150 MPa.
- We used a die of 12.5 mm diameter, which means an area of 122.65 square mm.
- Therefore the load or force which gives the optimum compaction is within the range,
- We used 2 batches and pressed the samples using different loads of 1, 1.5, 2, 2.5, 3 Tons. (Different batches indicate that it was prepared with the same raw materials, but at a different time.)
- For these samples, dimensional density, both green and sintered, were plotted against Log of pressure.

3.3 Characterisation

3.3.1 DENSITY MEASUREMENTS

Geometrical density was calculated using a digital vernier calliper. Firstly, the height and the diameter were noted down, 3 measurements for each dimension, to ensure accuracy. Volume was then calculated by the following formula:

$$Volume = \frac{\pi d^2 \mathrm{h}}{4}$$

Where d and h is the diameter and height respectively, both in cms.

For each pellet, mass was measured, and density was calculated by the following formula:

$$Density = \frac{Mass}{Volume}$$

Where mass is in gms, volume is in cubic cms. So, density is in gm/cc.

3.3.2 COMPRESSIVE STRENGTH MEASUREMENT

The maximum or yield force was measured by using a Tinius Olsen Materials Testing Machine (HK10S model). Then CCS was calculated by the following formula[2]:

$$Cold\ Compressive\ Strength = \frac{Maximum\ Force\ F}{Area\ on\ which\ the\ load\ was\ applied\ A}$$

In our case, we used pellets, therefore Area $A = \pi r^2$ in square mm where r is the radius in mm.

And maximum force given by the machine was in Newtons. So, CCS was in MPa.

3.3.3 DIAMETRAL COMPRESSION TEST

The yield force of the pellet was measured by a Tinius Olsen Materials Testing Machine (HK10S model). The Diametral Compression Strength was then calculated by the following formula[9]:

$$Diametral\ Compression\ Strength = 2.\frac{F}{(\pi.d.t)}$$

Where F is the maximum force in Newtons,

d is the diameter in mm and,

t is the thickness of our pellets in mm. So, Diametral compressive strength is in MPa.

3.3.4 SEM ANALYSIS

SEM was performed to check the compaction and density variation with the plasticiser amount. It was carried out at two magnifications, 900X and 2500X.

CHAPTER 4. RESULTS AND DISCUSSIONS

RESULTS AND DISCUSSIONS

The results of the experiments are as follows:

4.1. Optimization of dwell pressure

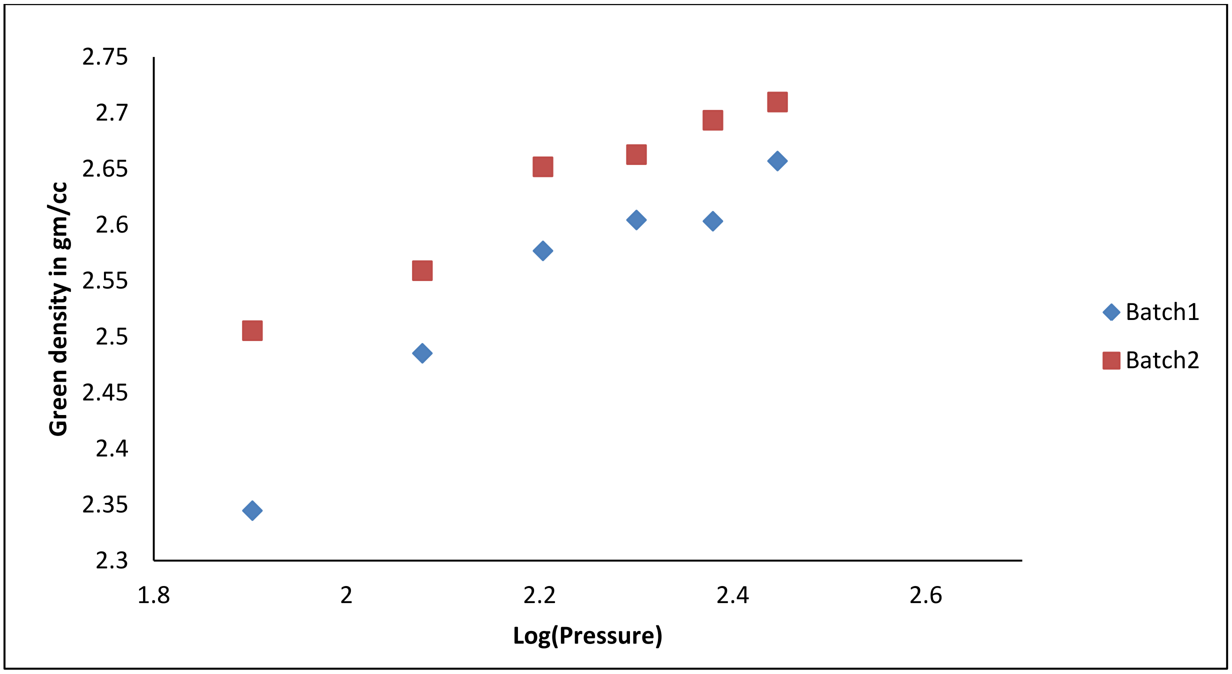

Fig.4.1(a) Variation of green density with log of forming pressure

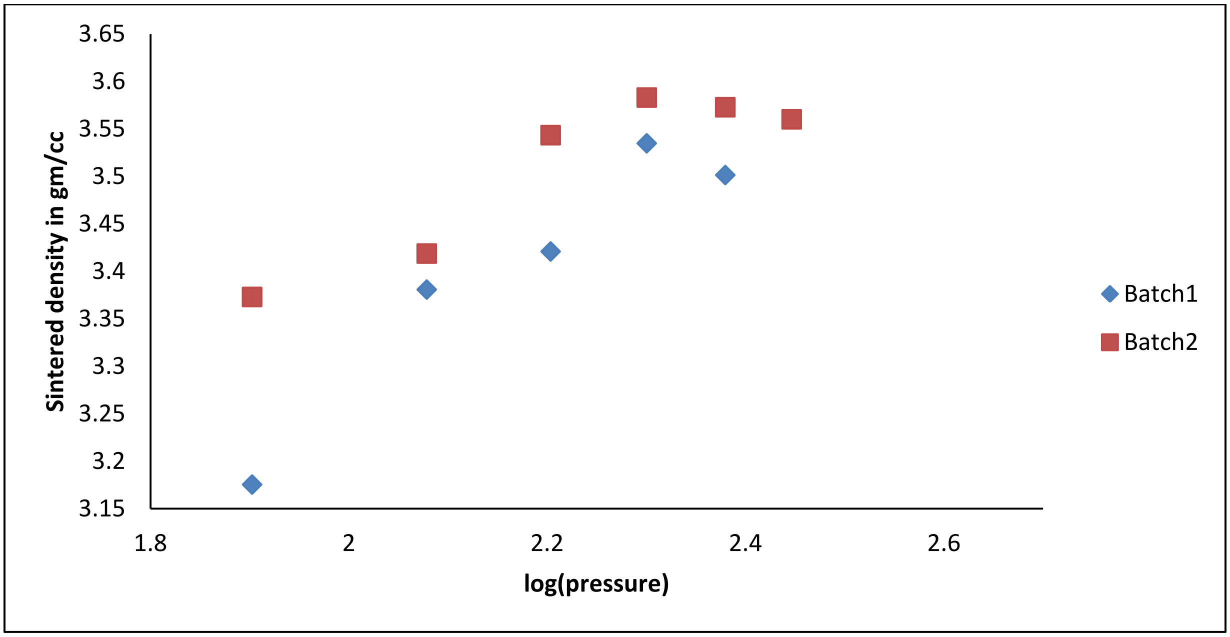

Fig.4.1(b) Variation of green density with log of forming pressure

Fig. 4.2 Bluish colouration (left-most) and pink colouration of the pellets (two on right) . Dark spots persisting even after firing (bottom) . Regions showing impurity incorporation in pellets.

From the Fig. 4.1(a), it could be seen that green density increased with the forming pressure. However, a point is expected in the graph, where any further increase in pressure doesn't improve the density and the graph becomes linear. From the Fig. 4.1(b), it was further clear that the highest sintered density is shown by the pellet pressed at 2.5 Tons and that it is the optimum dwell pressure for pressing.

When these pellets were placed in furnace, they were not covered and after the sintering process, they showed some defects described in Fig. 4.2. This could have been due to the diffusion of chromium (significant pink colour) already present in the furnace.

4.2. Effect of atmosphere on green and sintered density

In order to observe the effect of ambient atmosphere (temperature, humidity), two different batches were prepared, one pressed in December (RH=15%) and the other pressed in May (RH=30%).

The following 4 compositions were prepared and pressed in December 2011.

(a) Alumina + 5% PVA without PEG

(b) Alumina + 5% PVA + 0.5% PEG

(c) Alumina + 5% PVA + 1% PEG

(d) Alumina + 5% PVA + 1.5% PEG

The following 7 compositions were prepared in May 2012.

(a) Alumina + 5% PVA without PEG

(b) Alumina + 5% PVA + 0.25% PEG

(c) Alumina + 5% PVA + 0.5% PEG

(d) Alumina + 5% PVA + 0.75% PEG

(e) Alumina + 5% PVA + 1% PEG

(f) Alumina + 5% PVA + 1.25% PEG

(g) Alumina + 5% PVA + 1.5% PEG

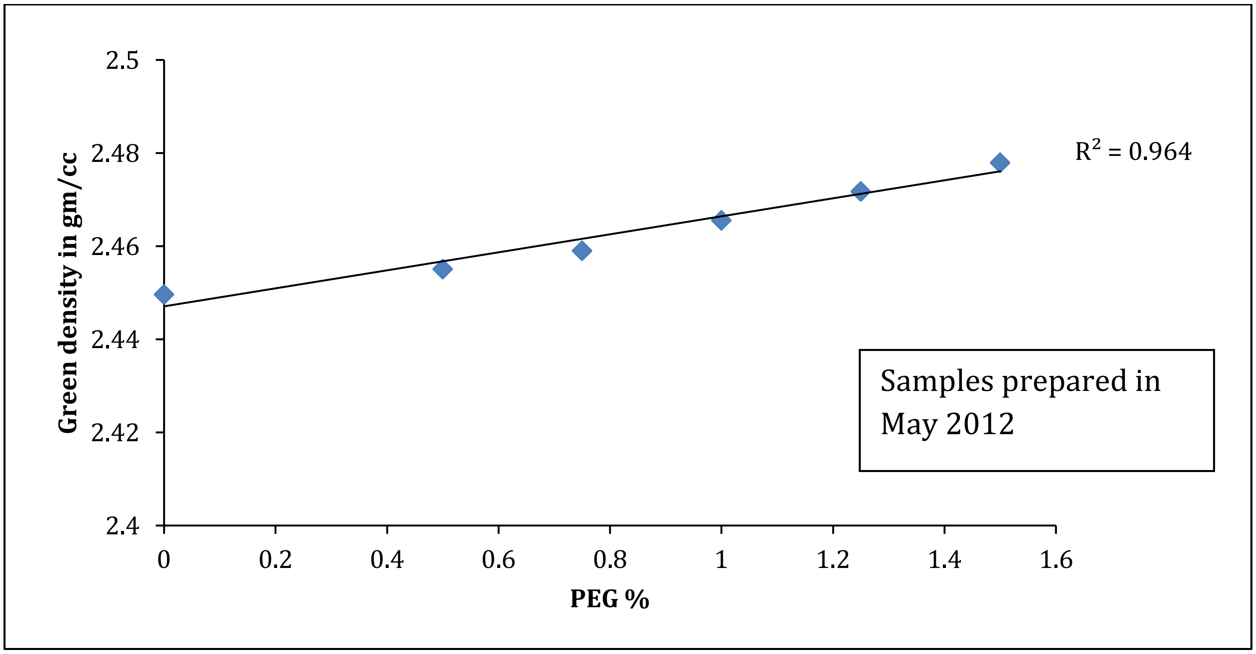

Fig.4.3(a) Variation of green density with PEG addition

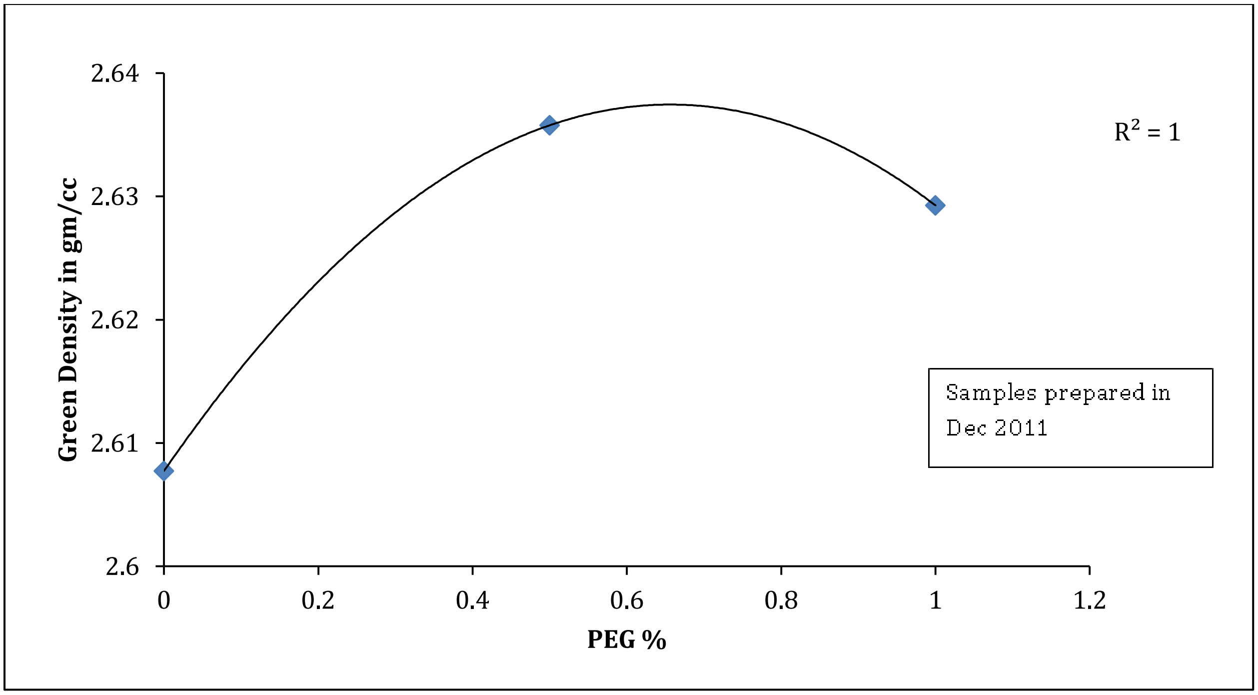

Fig.4.3(b) Variation of green density with PEG addition

Fig. 4.3(a) shows that for the samples pressed in December, the green density goes through a maximum and then decreases. However, for samples pressed in May 2012, the green density increases almost linearly with PEG content, Fig. 4.3(b). The difference in the green densification behaviour for the two sets of samples could be related to different plasticising effects of the binder.

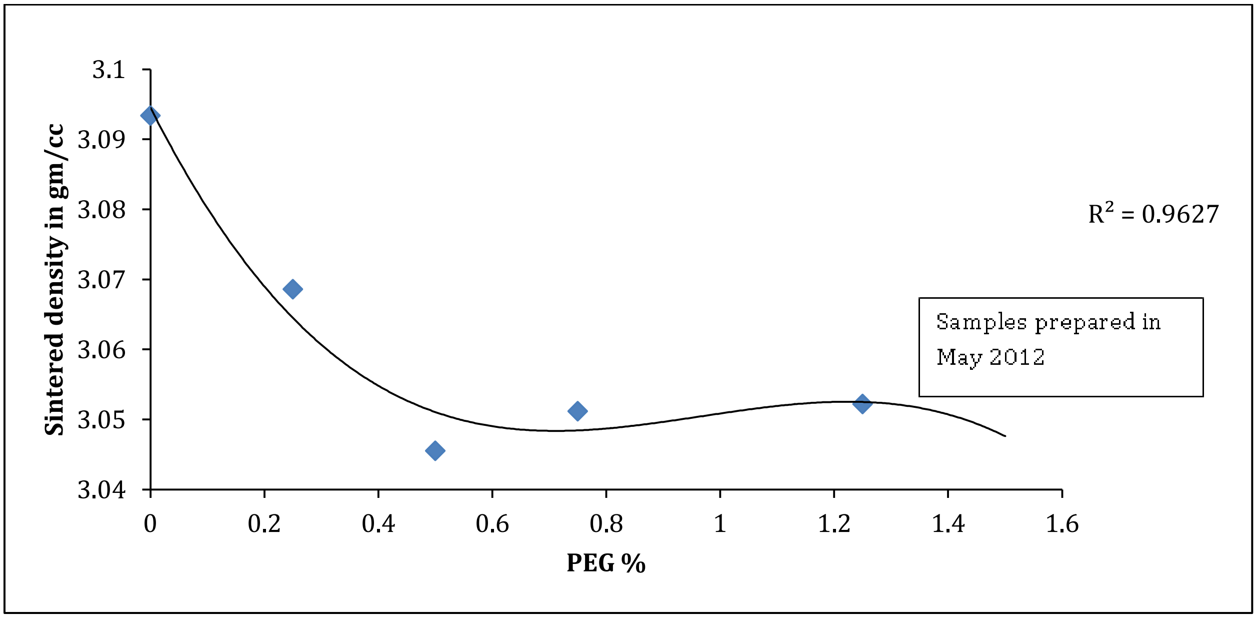

Fig.4.4(a) Variation of sintered density with addition of PEG (Alumina+5% PVA+PEG pellets sintered at 500°C/1 hour and 1600°C/2 hours)

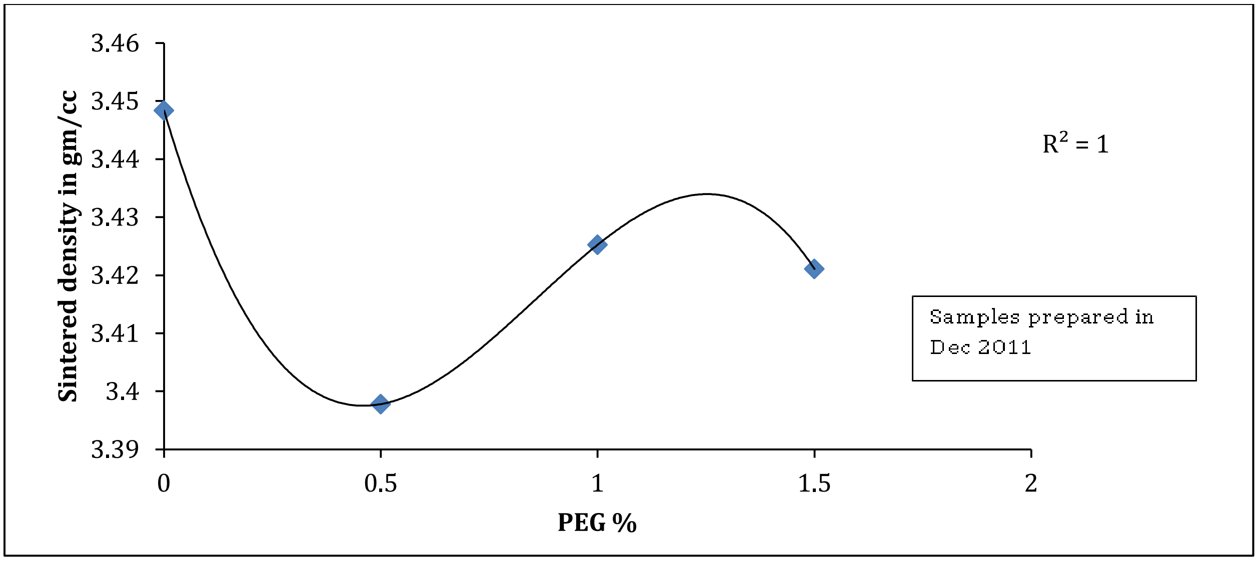

Fig.4.4(b) Variation of sintered density with addition of PEG (Alumina+5% PVA+PEG pellets sintered at 500°C/1 hour and 1600°C/2 hours)

Fig. 4.4(a) and Fig. 4.4(b) show the variation in the sintered density with PEG content. As seen from the graph, the sintered density decreases with PEG content and becomes constant from PEG content 0.5% onwards.

4.3. Strength of green samples

Green strength was measured in terms of Compressive Cold Strength.

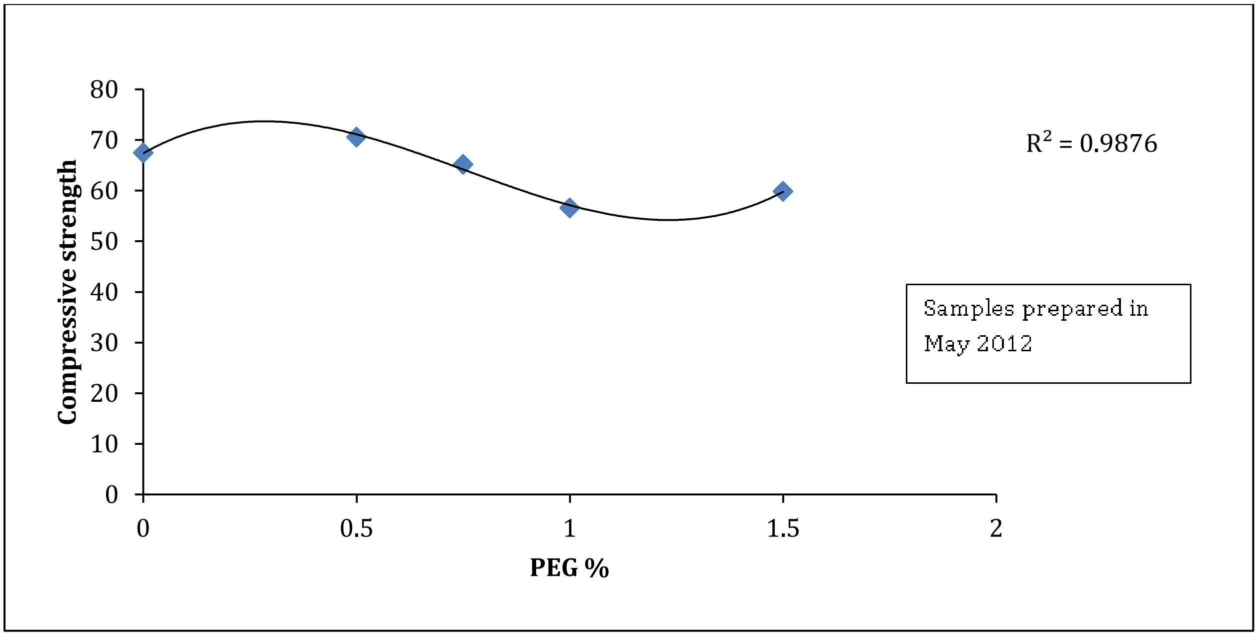

Fig. 4.5(a) Variation of green strength with addition of PEG

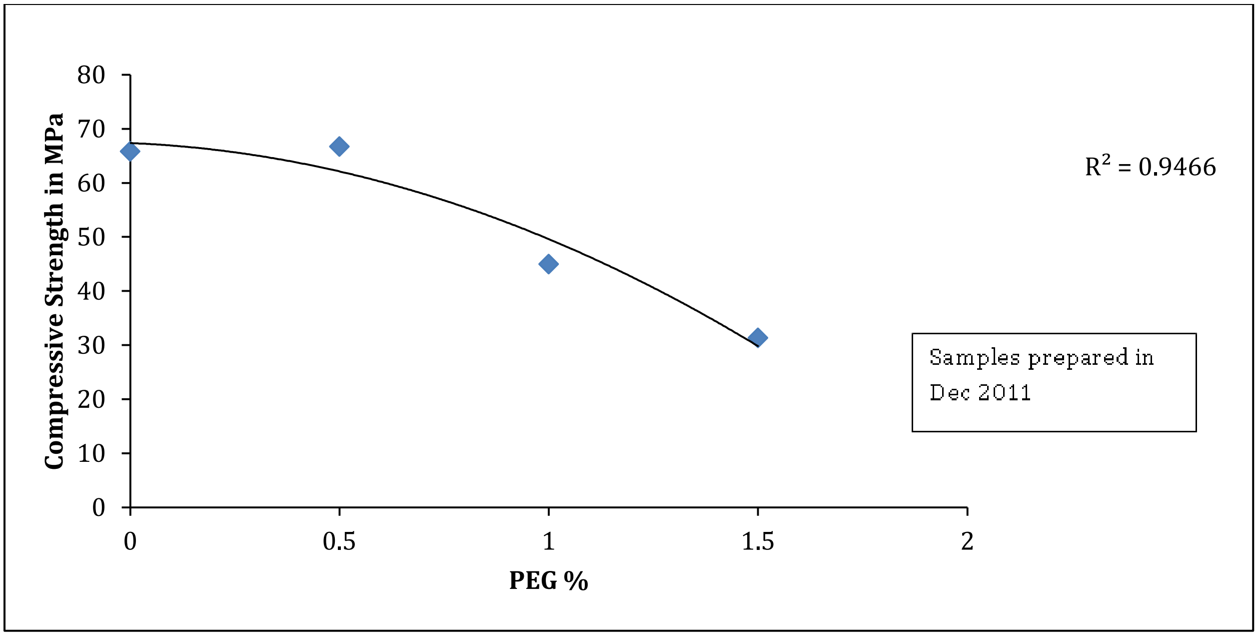

Fig. 4.5(b) Variation of green strength with addition of PEG

In the samples pressed in May 2012, the green strength for 0.75, 1 and 1.5% was around 70% but at 0.5%, green strength was the highest, 73%. However, for samples pressed in December 2011, a gradual decrease in green strength (from 68% to 40%) was observed with an increase in PEG content. The difference in the strengths could be due to different atmospheric conditions. In May, due to higher environmental temperature, the binder is more plasticised by the plasticisers but in December, both the binder and plasticiser are not effectively plasticised and therefore, at higher plasticiser content, the density decreases. But like May, the highest green strength was also observed at 0.5% PEG content. Thus, 0.5% PEG is optimum value of plasticisers.

4.4. Diametral Compressive Strength of sintered samples

Bi-axial Flexural Strength or Diametral Compressive Strength was measured on sintered samples. The CCS test could not be done on the sintered pellets because their CCS exceeded 10 KN, the highest load capacity of the machine.

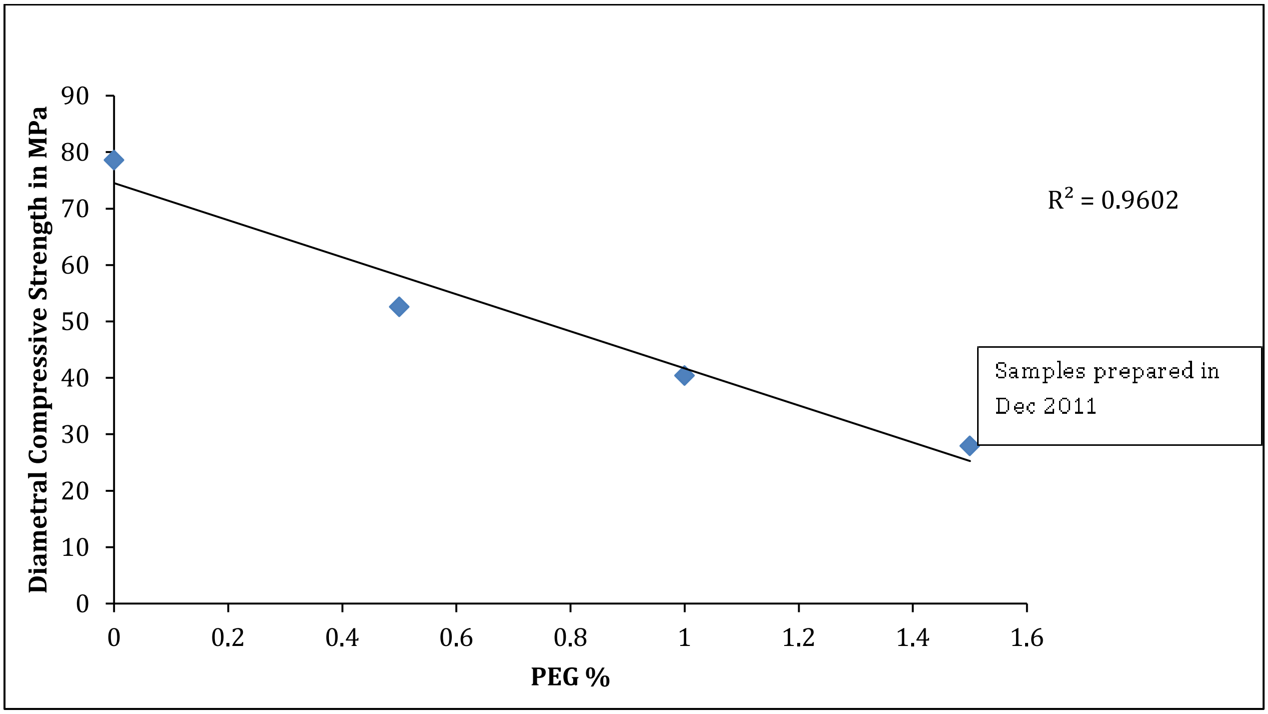

Fig.4.6(a) Variation of Diametral Compressive Strength of sintered pellets with addition of PEG.

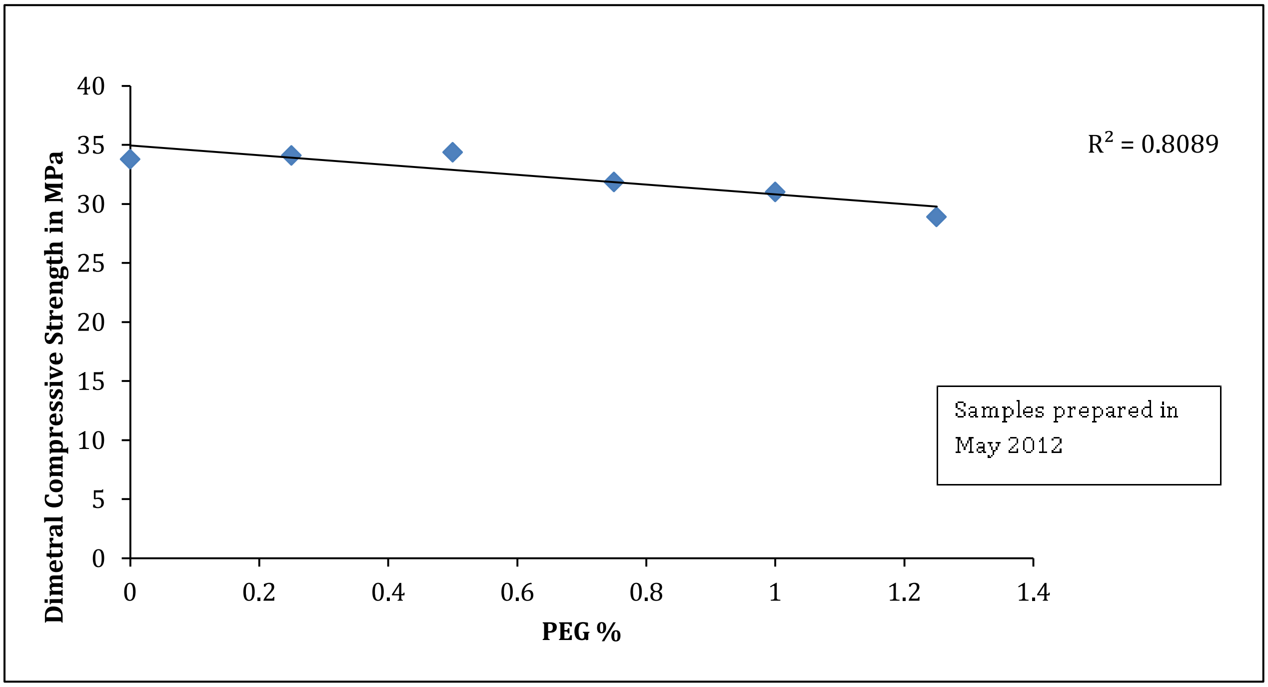

Fig.4.6(b) Variation of Diametral Compressive Strength of sintered pellets with addition of PEG.

The decrease in strength may be due to the difference in porosity level of the samples. Samples with higher PEG content had higher porosity due to the burnout of plasticiser. The decrease was less in May and more in December. This is again because of atmospherical effect. In May, the powder is well plasticised and pressing was better and uniform for all PEG additions. In December, due to poor plasticising effect on binder, the samples had higher and varying porosity after sintering. Hence the Diametral strength is decreasing sharply.

4.5. SEM analysis

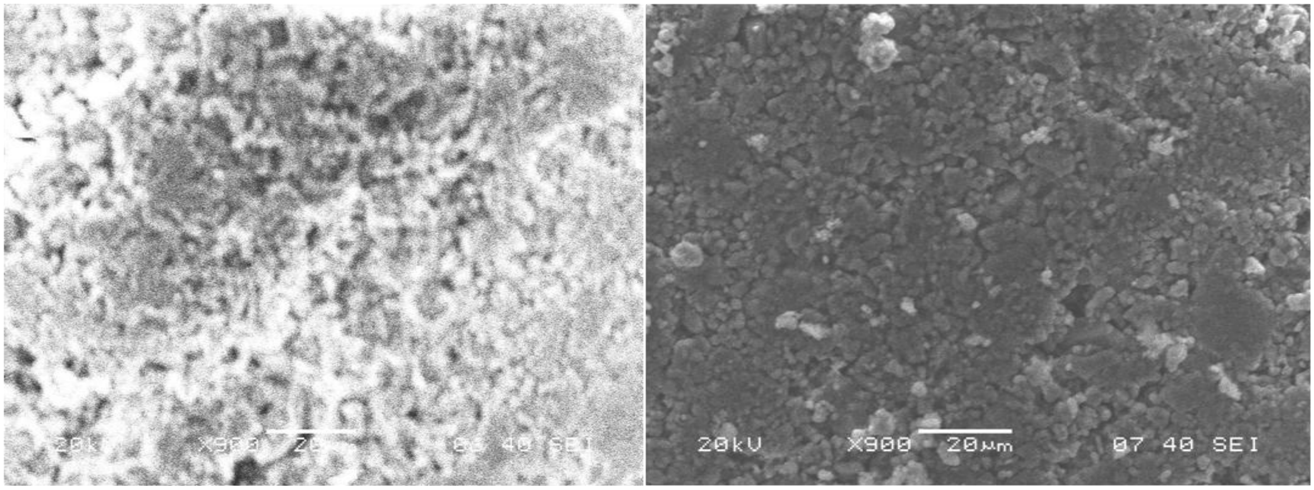

Fig.4.7. SEM micrographs at 900X magnification, LEFT(a): 0% PEG composition pellet; RIGHT(b): 0.75% PEG composition pellet.

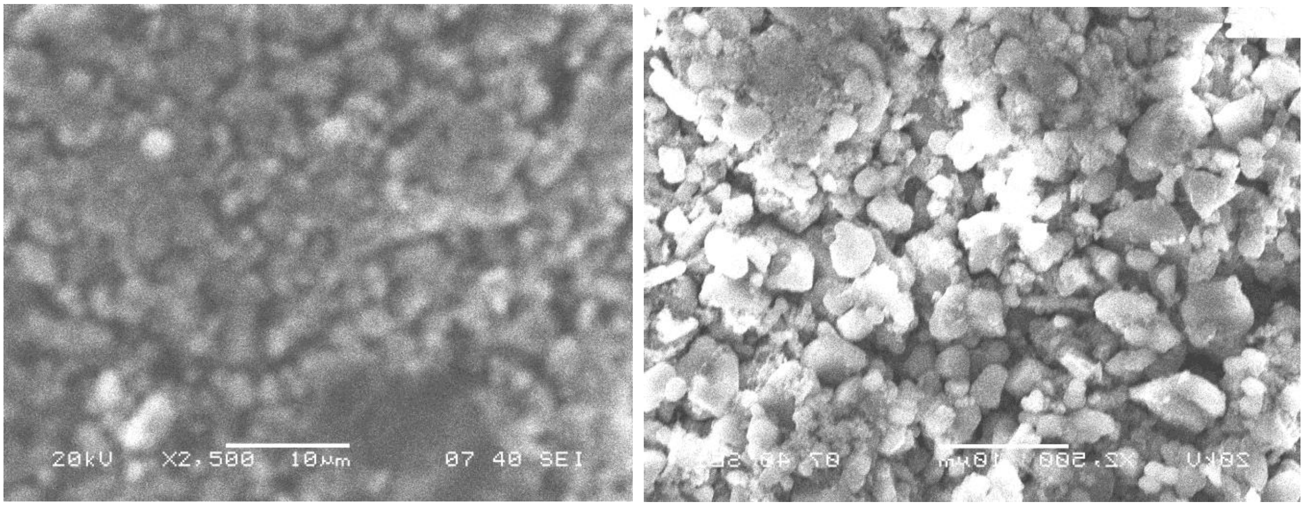

Fig.4.8. SEM micrographs at 2500X magnification, LEFT(a): 0% PEG composition pellet, RIGHT(b): 0.25% PEG composition pellet

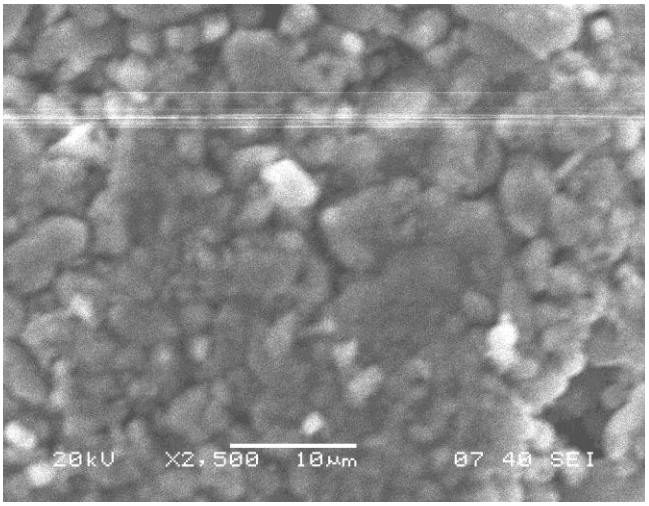

Fig.4.8.(c) SEM micrographs at 2500X magnification, 0.75% PEG composition pellet.

SEM micrographs show that with the increase in the plasticiser amount, the porosity decreased and the compactness and density increased.

CHAPTER 5. CONCLUSIONS

CONCLUSIONS

The following conclusions were drawn from this project:

1. The green density of the pellets improve with the addition of the plasticiser. However, a point is expected after which further addition leads to no further change, or even leads to a decrease in density. The lowest green density corresponds to the unplasticised one. There comes an optimum, and all the other densities displayed by other compositions are always lesser than this composition.

2. The sintered density show just the reverse trend. It decreases rapidly and then takes a more gradual slope, rather unaffected by further additions of the plasticiser. The highest sintered density is displayed by that pellet which was unplasticised, i.e. corresponding to the 0% PEG composition.

4. The highest green strength also has an optimum after which it decreases to give scattered values, still lesser than the optimum.

5. The sintered strength curve has an optimum at 0.25% PEG. This could be due to a competitive drive between the lowering of the sintered density due to the residue left after the PEG vaporises out and the increase in the green density because of the plasticising action. Later, the former factor becomes stronger and leads to a decrease in the overall sintered strength.

6. The atmosphere plays an important role in deciding the characteristics of the green product as well as the sintered. The ambient temperature, the humidity can affect the density to vary to an extent of 0.2-0.4 gm/cc.

CHAPTER 6. REFERENCES

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