EFFECT OF HOLDING TIME ON BINDER BURNOUT, DENSITY AND STRENGTH OF GREEN AND SINTERD ALUMINA SAMPLES

A Thesis Submitted

In Partial Fulfilment of the Requirement

For the degree of

BACHELOR OF TECHNOLOGY

By

DEBESH DAULAT MOHANTY

ROLL 107CRO28

TO THE

DEPARTMENT OF CERAMIC ENGINEERING

NATIONAL INSTITUTE OF TECHNOLOGY ROURKELA

MAY 2011

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Supervisor :

Prof. S. Bhattacharyya

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CERTIFICATE

This is certified that the work contained in the project entitled "EFFECT OF HOLDING TIME ON BINDER BURNOUT, DENSITY AND STRENGTH OF GREEN AND SINTERED ALUMINA SAMPLES" by Debesh Daulat Mohanty (Roll 107CR028) in partial fulfilment 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 him 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

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Debesh Daulat Mohanty

107CR028

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ABSTRACT

The present work deals with the effect of holding time on the binder burnout, bulk density and strength of the sintered alumina bodies. Binders provide better green strength to the ceramic bodies and their efficient removal from the system during sintering plays a crucial role in bulk density and strength of sintered bodies. The sintered bodies in this project were alumina samples which were prepared by dry pressing of granules. These granules were prepared by mixing reactive alumina (-75 micron) with binders solution of 3 and 4 weight percent. The thermal decomposition characteristics of the binders namely (Poly Vinyl Alcohol (PVA), Dextrin and starch) was carried out. On the basis of these studies, the binder burnout temperature was found to be 450 ^{0}C.Accordingly during sintering of the pellets a hold time was given at 450 ^{0}C. In order to see the effect of holding time on binder removal rate, the holding time was varied between- no holding (0 minute), 30 minute holding time, 60 minutes holding time. The samples were sintered at 1600 ^{0}C for one hour and the sintered samples were characterized for bulk density, apparent porosity, compressive strength and shrinkage. It was observed that Starch (3 %) added samples showed highest compressive strength and also had a high bulk density. It was observed that effective binder burnout could enhance strength of sintered bodies and incomplete removal may lead to defects like cracking, black coring, bloating and formation of closed pores which adversely affect the properties of the sintered bodies.

CONTENTS

Chapter 1

INTRODUCTION

1.1 Introduction

Ceramic processing is useful in the industrial production of a large variety of ceramic products. Shape forming processes are performed through various methods like:

1. Dry pressing
2. Extrusion
3. Injection Molding, etc.

Different types of binders are used in different quantities in these shape forming processes. A properly formulated and well controlled feed material is a key factor in pressing operations, choice of appropriate binders and their amount is of utmost importance.[1]. The binder in powder form is mixed with ceramic materials and then liquid is added to form a viscous binder solution in the extrusion process. Alternatively the binder may also be dissolved in suitable solvents which are then mixed with the powder. For injection molding, the binder solution is viscous and the binder amount is also high as the shaped products are very complex and thin walled.

1.2 Role of Binders

Binders have a variety of roles depending upon the forming process being used for shape forming of green ceramics. The major role of binders is to bind the granules together. This means that the binders improve the green strength of the products and help in handling of products before sintering. They can also act as plasticizers to provide plasticity to the system and also help to control the flow properties of the slurry. As a wetting agent, they help to improve the packing density in granules. [1]

Binders should enable production of crack-free green bodies at low amount of addition to promote clean pyrolysis. [2].

1.3 Classification of Binders

Organic binders are less expensive and therefore widely used. They provide better burnout at low temperatures and are efficient due to the absence of inorganic impurities. Inorganic binders are

used only when the inorganic component of the binder is compatible with the particle composition. [1]

Table 1: Classification of Binders [1]

Colloidal Particle Type	Organic Binder Microcrystalline cellulose	Inorganic Binder kaolin Ball Clay Bentonite
Molecular Type	Polyvinyl alcohol Starch, Dextrin, Paraffin	Sodium Silicates, Ethyl silicate

1.4 Widely Used Binders

1.4.1. Vinyl Binders

Poly Vinyl Alcohol is a water soluble binder and is manufactured by hydrolysis of Poly Vinyl acetate in presence of a catalyst. Fully hydrolyzed PVA contains less than 2% residual acetate and dissolves in hot water. [1]

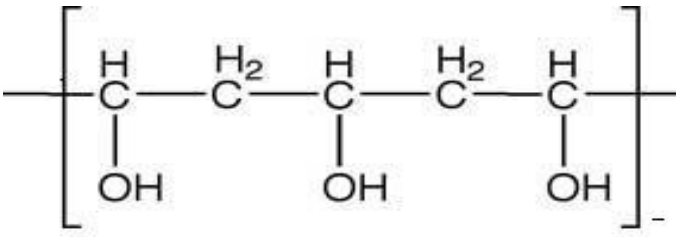

Fig.1: Molecular Structure of PVA [12]

It has C-C linkage and the -OH groups provide initial wetting and adhesion properties. PVA has strong affinity for adsorption on oxide particles dispersed in water. [1]. Poly vinyl butyral and poly-methacrylate binders dissolve in non-aqueous solvents also. PVA is largely used as a binder in dry pressed ceramics as it provides better mechanical strength to green bodies. [2]

1.4.2. Dextrin

Dextrin is a water soluble additive and can act as suitable plasticizers for aqueous and colloidal processing of alumina. [3].

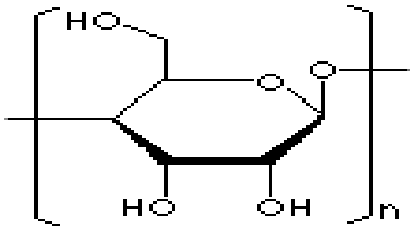

Fig. 2: Molecular Structure of Dextrin [13]

Dextrin is D-Glucose polymers of different molecular weights and is produced industrially by acid catalyzed hydrolysis or by the thermolysis of granular starch. [3].

1.4.3. Polyethylene Glycol Binders

Polyethylene Glycol (PEG) is polymerized ethylene oxide. It is commercially available in molecular weights ranging from 200 to about 8000 gm. /mol. Low molecular weight grades are relatively heat stable liquids. The PEG binders are very pure, water soluble and have limited solubility in various solvents. [1].

1.4.4. Film-Forming Binders

Paraffin derived from petroleum, candelilla derived from plants and beeswax of insect origin are some film-type binders. Paraffin is mixtures of straight-chain saturated hydrocarbons which tend to crystallize as pellets or needles. [1]. The mechanical properties of the plant waxes are related to secondary bonding between the molecules. [1].

1.4.5. Starch

Starch is a molecular type organic binder which is widely used because of their gelling ability in water. [4]. the seeds of cereal grains are the common sources from which starch is extracted for commercial use. [4].starch is a mixture of two polysaccharide types namely amylose which provides gelling properties and a branched type amylopectin. [4].

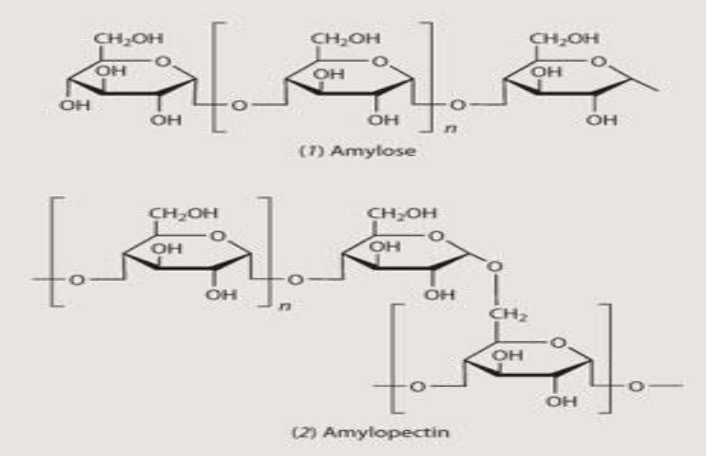

Fig. 3: Molecular Structure of Starch [14]

In the aqueous suspensions, the glucose units in starch give a strong hydrophilic character due to the exposure of large amount of hydroxyl groups present in it. [4]. Starch grains are white, dense and insoluble at the room temperatures. [5].

1.5 Plasticizers

A plasticizer is added to modify the viscoelastic properties of a condensed binder phase film on the particles. Binders used in ceramic processes must have good plasticity to be molded into different shapes. A water soluble binder like PVA is plasticized by water and relative humidity should be controlled to control its plasticizing effects. Water adsorbs on PVA and helps to reduce its Young's Modulus of Elasticity and increases the flexibility by providing greater elongation at rupture. But it reduces the strength of binders in the system. [1]. Organic liquids with a low vapor pressure than water are mostly used as primary plasticizers. Dextrin of molecular weights 6450-15,000 Daltons have been studied to show good plastic behavior in aqueous processing of alumina. [3].Water acts as

plasticizer for starch based products and its effects on the film formation depends on the amount of plasticizer used.

1.6 Dry Pressing

Dry Pressing is a very important fabrication process used to produce products of relatively high density. Proper formulation and controlling of feed material is a prerequisite for every pressing operation. [1].The feed material commonly used is a granulated powder and the binder that is added to the system gives better flow ability and packing density increases. Pressing eliminates large pores and product is uniformly dense and has strength enough to sustain ejection and handling. [1].The compact density of the granules depends upon the pressure applied and compaction takes place through three stages. [1].

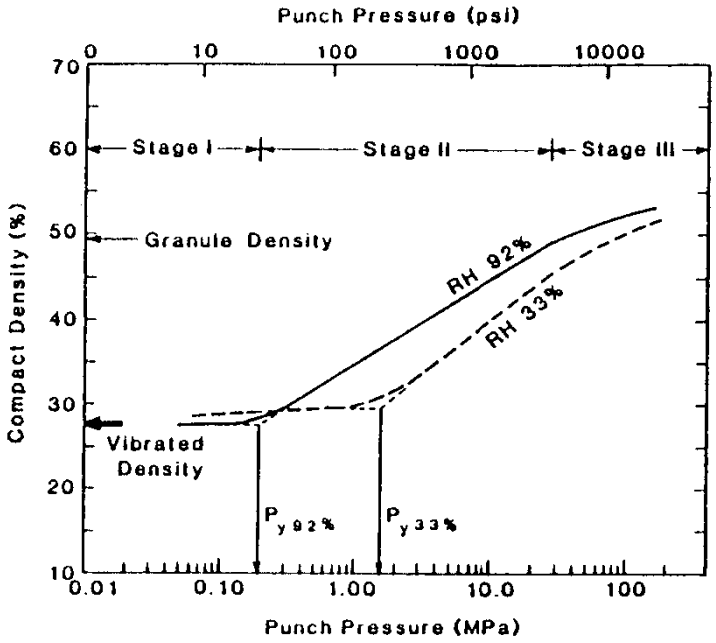

Fig. 4: Compact density of granules VS Punch Pressure. [1]

Addition of binders provides coating to granules and leads to sliding and rearrangement. The deformation reduces inter-granular porosity by increasing the area of contacts. [1].

The compact density of the system undergoes densification in three stages:

With a little increase in pressure, sliding and rearrangement causes initial densification. In the next stage, deformation of granules increases with increase in pressure beyond the yield pressure and the particles enter into neighboring spaces. The increased binder content and low amount of plasticizer are not suitable for densification as a greater pressure is required to achieve the required densities. Even higher densities are achieved by increasing of pressure in the next stage. The transition between different stages is not uniform and may occur simultaneously. [1].

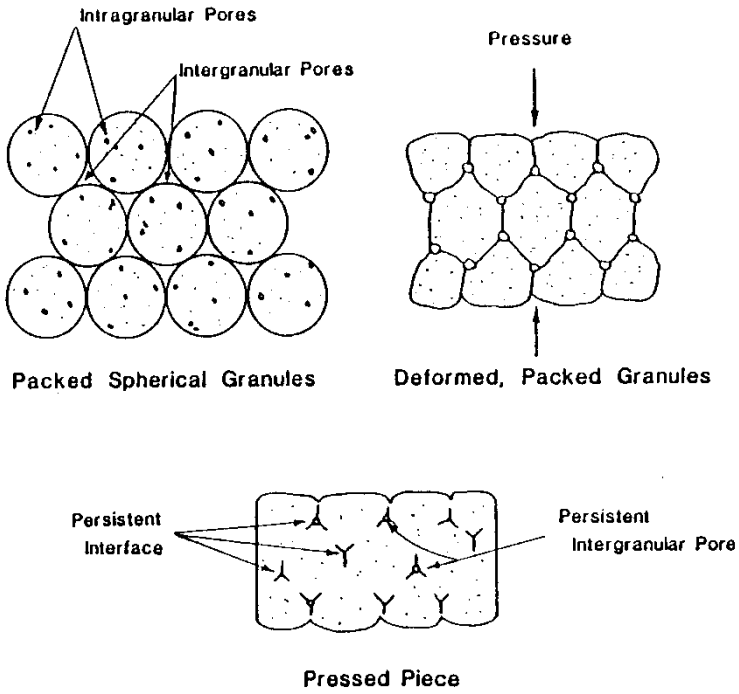

Fig. 5: Granule and Pore size change during Compaction Process [1]

Ejection from a die is an important process in formation of pellets. After compaction, the strength of the samples increase but poor ejection from mold can lead to defects such as cracking. Lubricants like stearic acids are used for proper ejection of samples. The spring back effect can be seen in the samples due to the elastic energies stored in them during compaction. A certain amount of spring

back is required for removal of sample from the die but excessive spring back may lead to various compaction defects. High amount of plasticizers reduce the spring back of a system. [1].

1.7 Effect of Binder Burnout

Binder burnout can be defined as the region of sintering where there is removal of binders, plasticizers, pore formers, dispersants and lubricants. They are removed by direct boiling off the organic compounds or by decomposition and combustion. The various parameters that have a marked impact on binder burnout include: [6]

1. Choice of binders and their amount used
2. Granulation and Mixing effects
3. Pressing Pressure
4. Holding Time and Heating Rates
5. Presence of Volatile Materials
6. Furnace Atmosphere

Although binders enhance the green strength of the bodies its effective removal has a huge impact on the sintered density, apparent porosity and compressive strength of the sintered bodies. Sintering is the rate limiting step of every industrial process and its regulation can control the properties of the fired body. [6].The binder has to be completely removed from the products during the high temperature processes. In the event of incomplete removal of binders, the sintered bodies will have defects like- black coring, sealed pores and bloating. This may lead to variation in pore size and shapes and the distribution of pores. Bubble formation in green bodies during binder decomposition can also lead to several defects within a fired body. [7].

1.8 Steps for effective Burnout

As binder burnout plays such a crucial role in the final properties of sintered bodies, efforts should be taken to make the process effective. The optimization of binders for different processes can prove to be a profitable venture for the industries. [6].The steps include:

1. DSC-TG analysis of binders
2. FTIR analysis of their off gas behavior

The DSC-TG analysis helps to view the weight loss behavior of the binders at a particular temperature range.

FTIR analysis shows the chemistry behind the weight loss and helps to determine the gases that were given out during the burnout. It also gives an indication about the nature of bonds and related compounds and how they are getting removed.

After determining the temperature range where there is maximum weight loss, the logical way is to heat at slow heating rates at that temperature with some holding time to ensure complete removal of the binders. Low temperature holds have been very effective in reducing cracking as the analysis has thrown light upon the exact temperature for dehydration and removal of organic binders. [6].

There have not been a large number of researches on the effect of binder burnout on the mechanical strength of the fired bodies and with this background the present study proposes to investigate the effectiveness of different binder types as well as their amount on the binder burnout characterization as well as on the strength and density of sintered bodies.

CHAPTER 2

LITERATURE REVIEW

Sikora et al. [2] studied the plasticity of water soluble dextrin for aqueous colloidal processing of alumina and found that the fluid behavior of dextrin is highest at concentrations between 3 and 5 weight per cent. The thermal decomposition of dextrin produces mainly water and carbon dioxide and the decomposition products could be removed at low temperature which prevented cracking of the fired bodies. It was also observed that Daltons (Molecular Weight > 15,000) had poor plasticity.

Baklouti et al. [3] studied the binder burnout and mechanical strength of dry pressed ceramics containing Poly Vinyl Alcohol as binder and concluded that water acts as a plasticizer for Poly Vinyl Alcohol (PVA) and that strength of ceramics increases on binder addition. He also concluded that mechanical property measurement of green pressed should be carried out immediately after ejection from the die. The density and strength of ceramic were dependent were dependent on binder properties.

The microstructure of fractured sample showed that inter-granular fracture taking place between deformed granules and the fracture process leads to micro-crack generation in the samples. Inner part of the compact has less concentration of binder and therefore the core of the granule may act as critical defect. They noticed a significant decrease in strength of samples having Poly Vinyl Alcohol (PVA) as binder upon drying. They have assumed it to be the effect of the removal of residual moisture as residual moisture gives better plasticity to PVA and improves its strength.

Liang et al. [8] demonstrated that binder diffusion coefficients of PVA were dependent on specimen size, solution volume and temperature. Micro-structural changes during drying were analyzed to find the relation of binder diffusion with these parameters.

Zhang et al. [9] investigated various mechanisms of binder segregation in aqueous alumina - poly vinyl alcohol suspensions. Concentration of PVA and drying temperature had marked effect on segregation of binders. He suggested that surface segregation resulted from interaction between the migrated liquid and the diffusion of polymers.

Mandanas et al. [10] summarized the relationship between binder type and distribution by conducting experiments on 4 wt. % and 2 wt. % PVA suspension with reactive alumina and concluded that binder segregation is a function of permeability and depended on particle network and binder distribution in a body.

Lyckfeldt et al. [5] mixed starch with water and heated the suspension up to 60-80 C which resulted in the swelling of starch particle due to the uptake of water. The swelled starch acted as binder for the consolidated body and helped in demoulding. Studying the processing of thermal insulation materials with controlled porosity indicated that starch had good gelling properties in water and were easy to burn out which made them environment friendly. At larger amounts of native starch, the unstable properties may lead to a higher degree of breakdown of the granules during the water processing resulting in higher degree of shrinkage of the alumina matrix.

Niesz et al. [11] studied the strength characterization of powder aggregates and concluded that the microstructural developments and various properties of the system depended on strength of aggregates and could be best studied by relating the pressure density data. Reactive alumina was used for this purpose as they can have fine particle sizes of different grades.

Mitchell et al. [6] studied the importance of binder burnout and the instrumentation for effective analysis of burnout on sintering. They found out that the burnout behavior was affected by pellet

size and weight. The instrumentation helped to close the gap between the samples studied industrially and in laboratories.

Paik et al. [12] studied the importance of complete binder removal in Multilayer Ceramic Capacitor and studied the pore size distribution. The conditions of sintering and the temperature affected the microstructural properties of the system during binder burnout with no effects of heating rates on binder removal.

CHAPTER 3

EXPERIMENTAL WORK

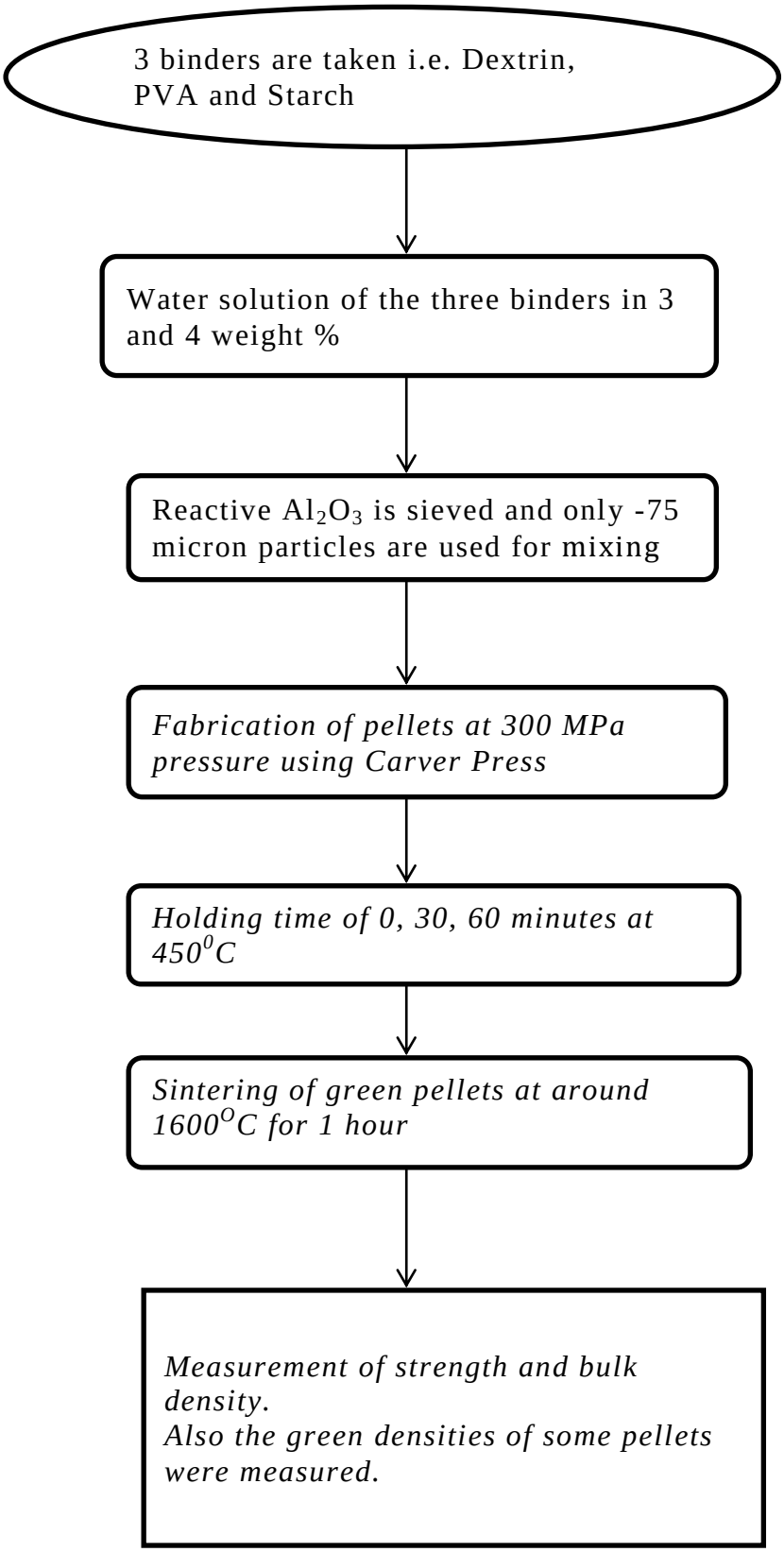

Fig. 6: Flow Chart of the Plan of work

3.1 Fabrication of Pellets

3.1.1 Preparation of Binder Solutions

Three different binders: Poly Vinyl Alcohol (PVA), Dextrin, Starch was used for preparation of binder solution.

Table 2: Binder Specifications

Binder Powder	Source
Poly Vinyl Alcohol(PVA)	LOBA Chemie
Starch	LOBA Chemie $(C_6H_{10}O_6)n$
Dextrin	LOBA Chemie $(C_6M_{10}O_5)n$ X H_2O

Dextrin Solution

In a 250 ml beaker, 3 or 4 grams of binder is taken and added to 100 ml of water and mixed to form 3 and 4 weight per cent of Dextrin solution.

Poly Vinyl Alcohol Solution

In a 250 ml beaker, 100 ml of distilled water is taken and heated at a constant rate for 20 minutes on a hot plate. A magnetic stirrer was added into the beaker and Poly Vinyl Alcohol was added in batches and mixed thoroughly to form 3 and 4 weight per cent of PVA solution.

Starch Solution

3 and 4 grams of starch powder were weighed and added separately in a 250 ml beaker containing 100 ml of distilled water. Stirring for few minutes gave starch solution of 3 and 4 weight per cent.

3.1.2 Sieving of Reactive Alumina

Reactive alumina of different particle size distribution was taken. The reactive alumina from Almatis was sieved in a sieve-shaker. Only the (-75 micron) sized particles were used for the experimental purpose.

3.1.3 Formation of Slurry on Mixing

Addition of binder solution to the reactive alumina in optimum quantity was followed by mixing in the mortar pestle and subsequent drying under the IR lamp. Then after drying the granulated powder obtained was made finer by further crushing and a certain amount of powder was obtained for preparing of pellets.

3.1.4 Dry Pressing by Using Carver Press

The powder samples were pressed using a circular metallic die of dimensions (12.50 mm diameter) in the Carver press (CARVER Co. U.S.A.). 3 % Stearic Acid was used as lubricant for smooth movements of die and acetone was used for cleaning the die. A pressure of nearly 300 MPa was applied on every sample to form a pellet of average dimensions (12.45mm * 2.45mm).The force used for pressing of powder = 3.5 US Tons

1 US Ton = 31,137.54 KN

3.2 Sintering of Pellets

The green pellets were sintered in the electrical resistance heating furnace with $MoSi_2$ heating element (Prysalch & Co. Kolkata) at 3 ^{0}C/minute heating rate. The sintering temperature was fixed to be 1600 ^{0}C and holding time at this temperature was 1 hour. Each batch of pellets had 5 samples for respective binder solution. Upon determining the maximum weight loss to be at 400-450 ^{0}C temperature range, the heating rate till 450 ^{0}C was slowed to 2^{0}C/minute and different holding time were given for different batches.

Different batches were prepared using the three types of binders and at different wt. %.

Batch 1- Green Bodies

Batch 2- 2 ^{0}C/ minute to 450 ^{0}C at no holding time

Batch 3- 2 ^{0}C/ minute to 450 ^{0}C at 30 minutes holding time

Batch 4- 2 ^{0}C/ minute to 450 ^{0}C at 60 minutes holding time

After sintering, the characterization of sintered bodies for different properties was done.

3.3 Measurement of Bulk Density and Apparent Porosity

Bulk density and apparent porosity of sinter specimens were determined by Archimedes principle. Sintered samples were weighed in dry state. Samples were immersed in kerosene and kept under a vacuum desiccator 4 hours to ensure that kerosene filled up the open pores completely. Then, soaked and suspended weights were measured.

The apparent porosity and bulk density were calculated as follows:

W_d =Dry weight of the sample,

W_s = Soaked weight of the sample,

W_a =Suspended weight of the sample

$$\text{Bulk density} = \frac{W_d}{W_s - W_a} * \text{Density of kerosene}$$

$$\text{Apparent porosity} = \frac{W_s - W_d}{W_s - W_a} * 100$$

Density of Kerosene = 0.79 gm. / cm^3

3.4 Measurement of Compressive Strength

Compressive strength of green bodies and sintered bodies were calculated using the Testing Machine (Tinius Olsen).It is a press used for circular pellets with a range of 10 KN and it measures the maximum force on a pellet and also its compressive strength. The samples were kept towards the center and once the operation started the force kept on increasing until the sample cracked and the force decreased considerably to remain constant after some time.

The compressive strength determined was of two types:

1. Biaxial Flexural Strength

2. Compressive Strength

For few batches, Biaxial Flexural Strength was measured. For this measurement the pellets were kept diametrically under the load.

Biaxial Flexural Strength = 2*Force/ (3.14 * Diameter * Thickness)

After analyzing of data, it was difficult to comprehend the relation between the binders and the compressive strength. So the samples were tested for compressive strength where the samples were placed normally between the loads.

Compressive strength helped to get a better understanding of the samples and the formula used was: Compressive Strength = Force/ 0.785 * $(Diameter)^2$

The factor 0.785 is for the (3.14/ 4).

CHAPTER 4

RESULTS AND DISCUSSION

The experiments under this project were performed to determine the exact relations between the binder burnout and the mechanical strengths of the sintered bodies.

4.1 Batch Optimization of Green Bodies

4.1.1 Determination of mass of powder for preparation of pellets

Density = Mass/ Volume

Expected Dimensions of a single sample is (12.50 mm * 3.0 mm)

Volume of the disc = 3.14 * (diameter)^2 * (Thickness)/ 4 = 0.368 cm^3

Density of Reactive Alumina = 3.90 gm. /cm^3

Mass of the sample = Density * Volume = 3.90 * 0.368 = 1.43 grams

But in random packing, only 52 % solid density is achieved and hence there is 48 % of void spaces.

Therefore, the mass of the sample = 52 % of 1.43 grams = 0.74 grams

4.1.2 Amount of Effective Binder in the Batch

For preparing 5 or 6 samples 4.5 grams of reactive alumina powder was used. For 4.5 grams of reactive alumina 4.5 ml of binder solution was added.

Case 1:100 ml of solution contains 3 grams of binder

1.5 ml of solution contains 0.135 grams of binder

Percentage of binder in the batch is (0.135/ 4.5) * 100 = 3.4 %

This means that even if binder solution has its strength as 3 wt. %, effective binder present in the batches is different and changes with the amount of binder solution added to form slurry.

Case 2: 100 ml of solution contains 4 grams of binder

4.5 ml of solution contains 0.18 grams of binder

Percentage of binder in the batch is (0.18/4.5)* 100 = 4.5%

This is similar to case 1 as the effective binder in the batch is more than 4 wt. %.

4.2 Choices of Binders

For this purpose first three different binders were chosen namely:

1. Poly Vinyl Alcohol (PVA)
2. Dextrin
3. Starch

The reasons for using these binders are:

2. They are easily available and widely used industrially.
3. They are water soluble
4. They are less toxic
5. They all have low burnout temperature

4.3 Thermal decomposition of binders

DSC- TG analysis for the three binders was carried out by other fellow investigators and temperature for maximum weight loss was determined for the three binders. Binders used for this purpose were in form of powders. The DSC/ TG experiments were conducted in a Netzsch 449C Thermal Analyser. The samples were heated in flowing Argon atmosphere at a heating rate of 5ºC/min. The weight loss measurements were also done in the same instrument and the results are shown in Figures.

1. Poly Vinyl Alcohol (PVA)

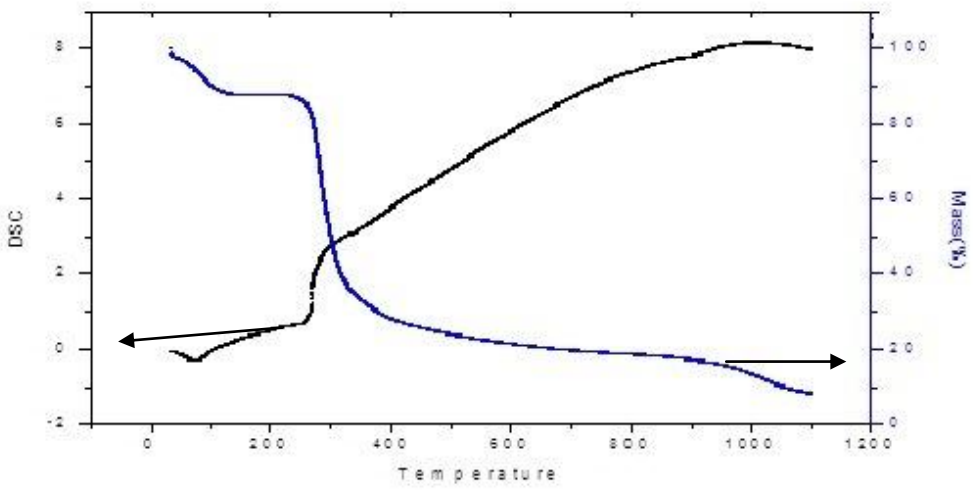

Fig. 7: DSC-TG of Poly Vinyl Alcohol (PVA)

Three very distinct endothermic peaks at around 100°C, 230°C and 260°C are observed for PVA. A broad exothermic peak is seen at around 740°C. The maximum weight loss is around 90% of the original sample mass and takes place at around 400ºC.

2. **Dextrin**

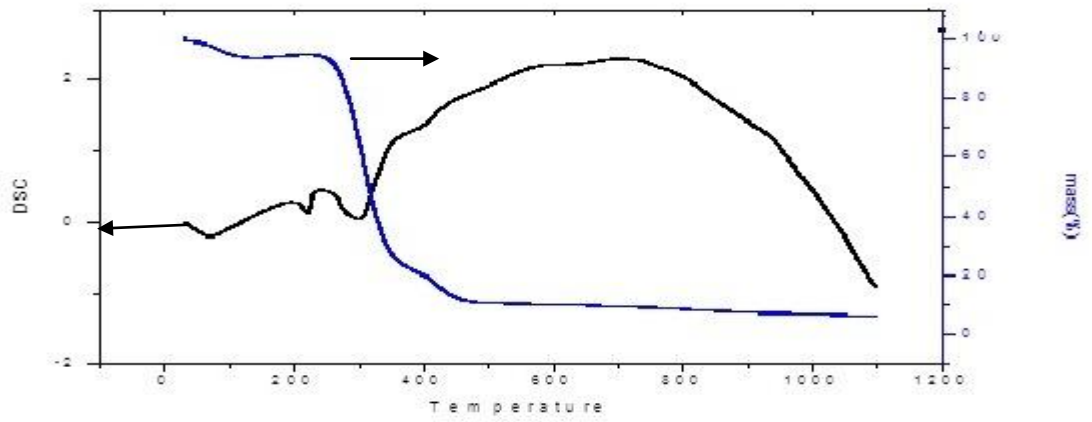

Fig. 8: DSC-TG of Dextrin

DSC/TG plot of dextrin at 5°C/min heating rate shows a broad endothermic peak at around 100ºC and another endothermic peak at 260°C. TG curve of 5ºC/min heating rate shows that the approximate temperature at which there is maximum weight loss is 360°C.

3. Starch

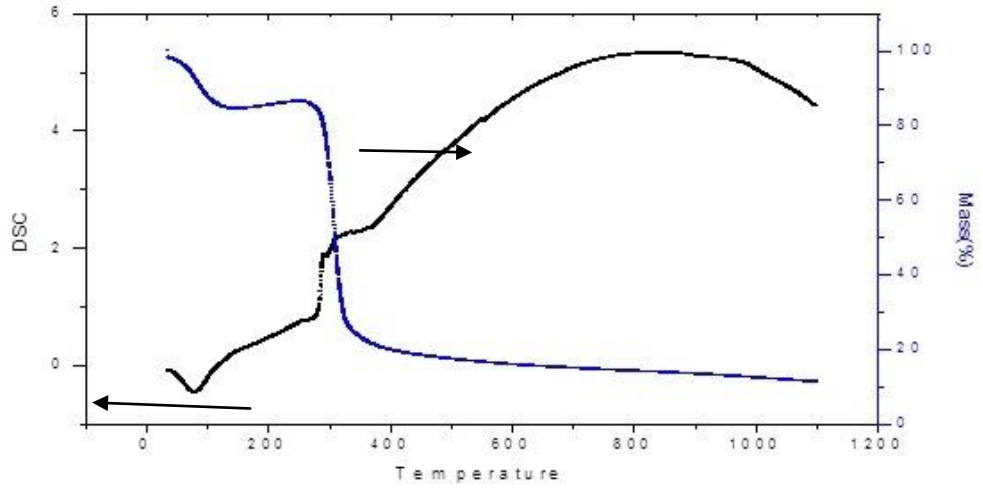

Fig. 9: DSC-TG of Starch

For starch distinct endothermic peaks at around 80°C, 280°C and 310°C are observed at 5°C/min heating rate. A broad exothermic peak is seen at around 880°C. Maximum weight loss takes place at around 380 °C and final residue remaining is 10% of the original mass. The weight loss behavior corresponds to DSC curve at 80°C and 310°C. The weight loss is seen due to removal of moisture and decomposition of carbohydrates.

The DSC-TG analysis showed that the maximum weight loss for binders was around 400- 450 ^{0}C. This is the reason of having additional holding time at 450 ^{0}C while sintering of the samples.

4.4 Batch 1 Analysis

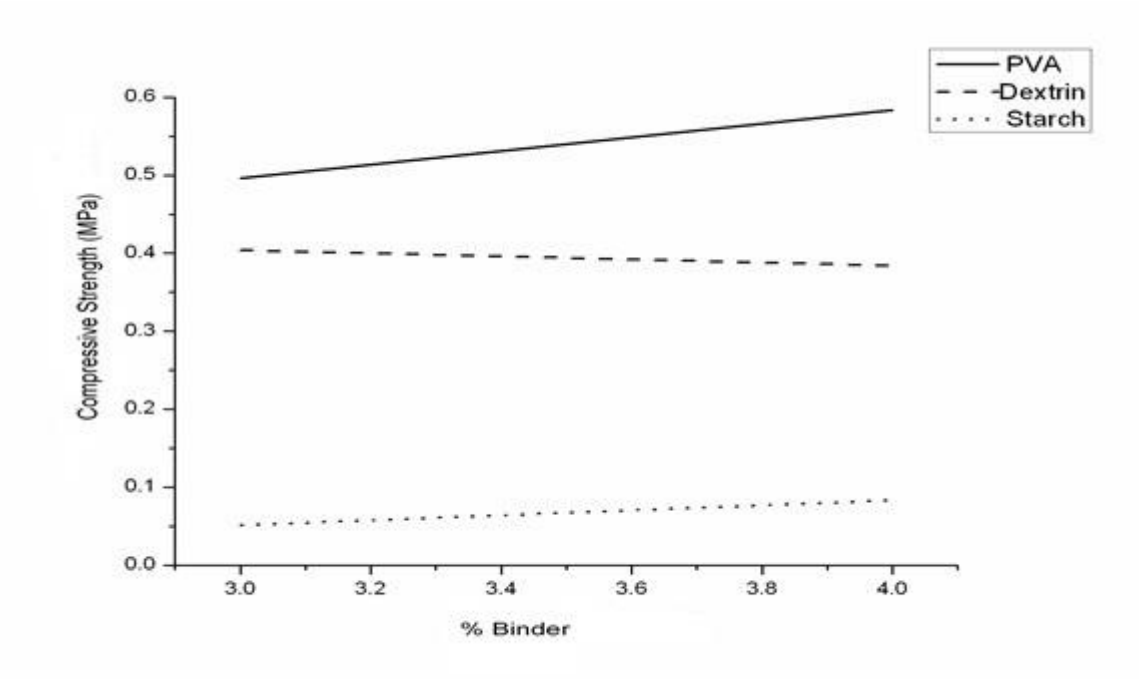

Fig.10: Compressive strength (MPa) of green pellets VS binder percentage

The green bodies after pressing were tested in diametral compression in a Material Testing Machine (Tinius Olsen). The results are summarized below.

The compressive strength of the 3 and 4 weight per cent of all binders added samples showed that PVA added samples had the maximum strength while the starch added samples had minimum strength. The reasons for this behavior of Poly Vinyl Alcohol (PVA) added samples may be due to the fact that water gives better plasticity when mixed with PVA. [3]. The PVA mixed slurry was viscous and can be a possible reason for higher strength. Starch mixed slurry had low viscosity and therefore low compressive strength. Starch is mainly used in porous ceramics and thus scores low in mechanical strength. [5].

Batch 2

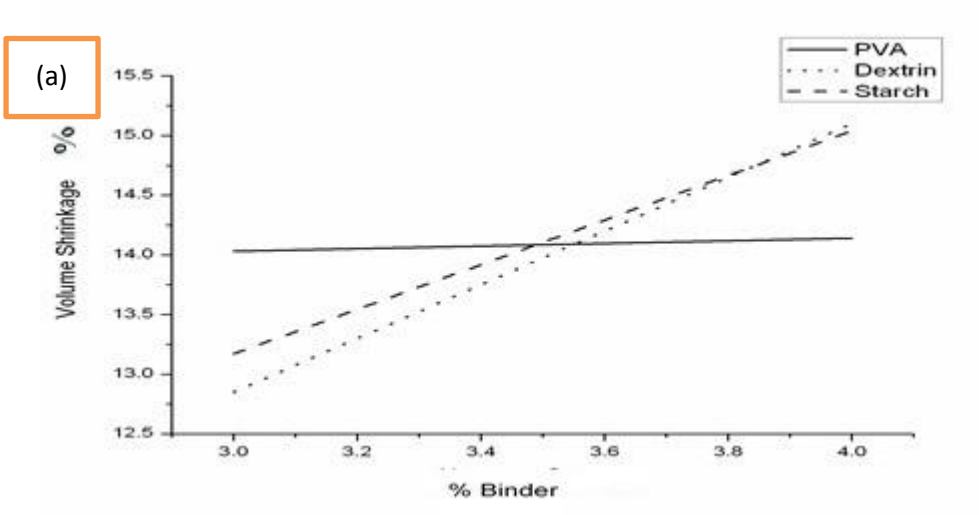

Fig. 11(a): Volume Shrinkage VS Binder Percentage

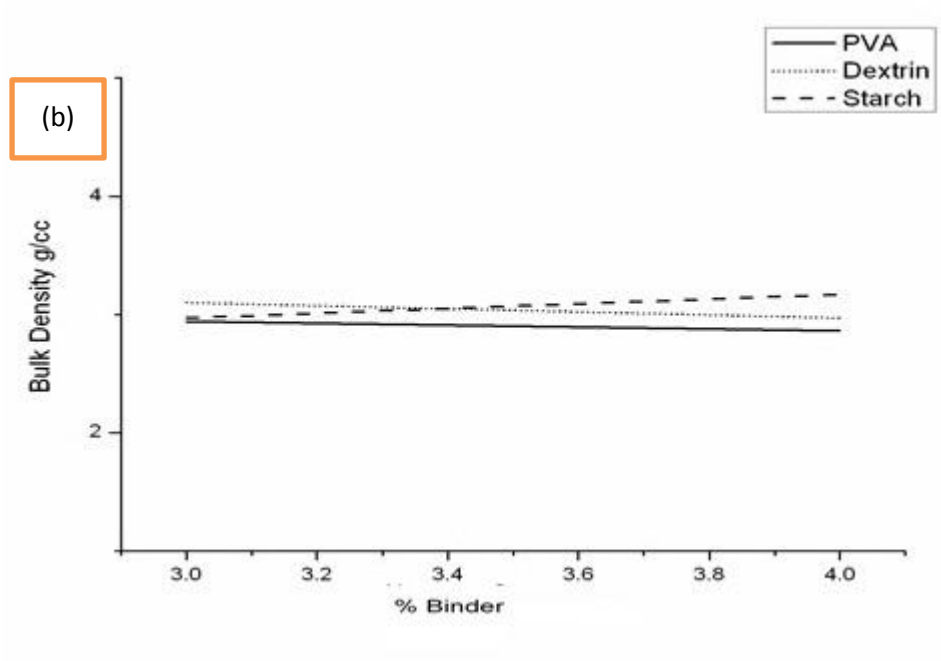

Fig. 11(b): Bulk Density VS Binder Percentage

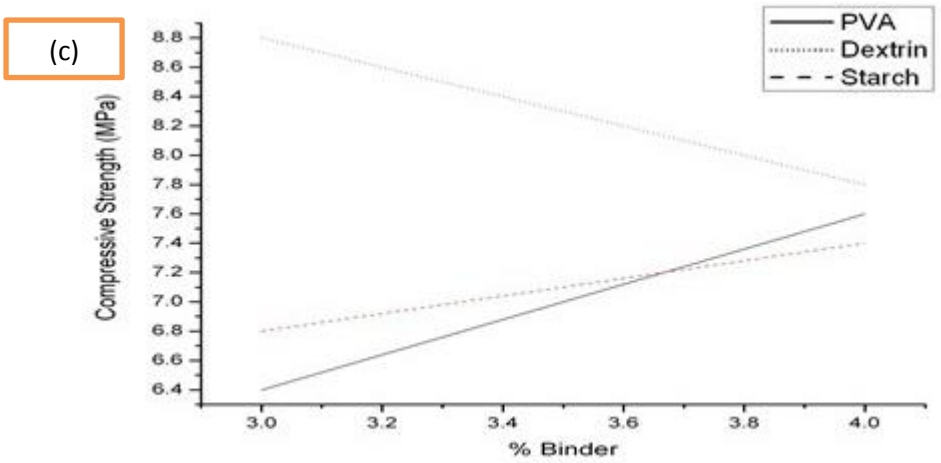

Fig. 11(c): Compressive Strength (MPa) VS Binder Percentage

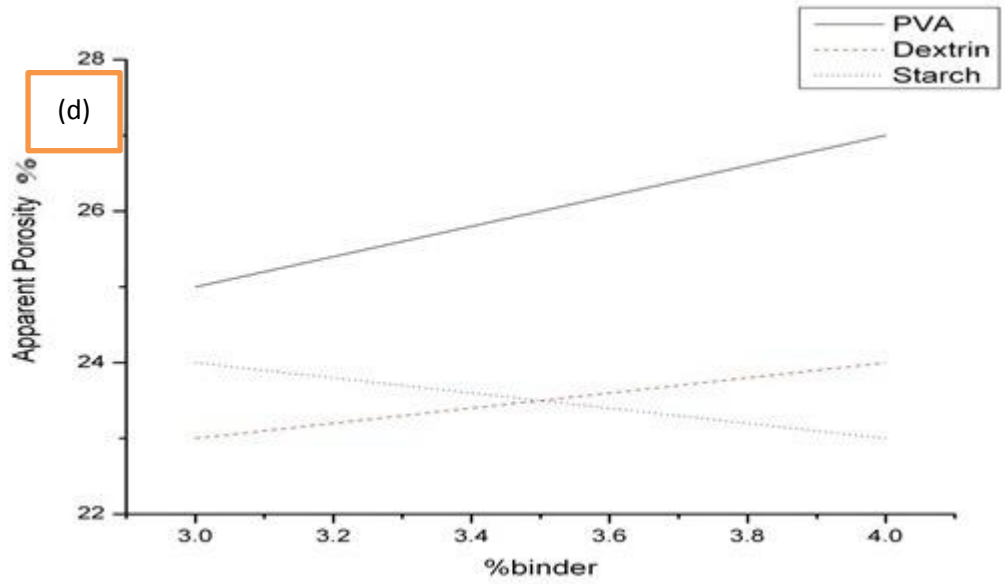

Fig. 11(d): Apparent Porosity VS Binder Percentage

4.5 Batch 2 Analysis

For Batch 2, the samples were heated slowly till 450 ^{0}C at 2 ^{0}C/ minute with no holding time. The graphs show that:

1. For PVA and Dextrin added samples, the strength increased with increase of binder content.

2. The compressive strength of Starch (3 %) added samples were found to show maximum compressive strength.

3. No huge difference was observed in bulk density and apparent porosity of the samples tested.

4. Starch added samples had better compressive strength after sintering because the binder burnout of starch binder was effective than other binder added samples.

Batch 3

a

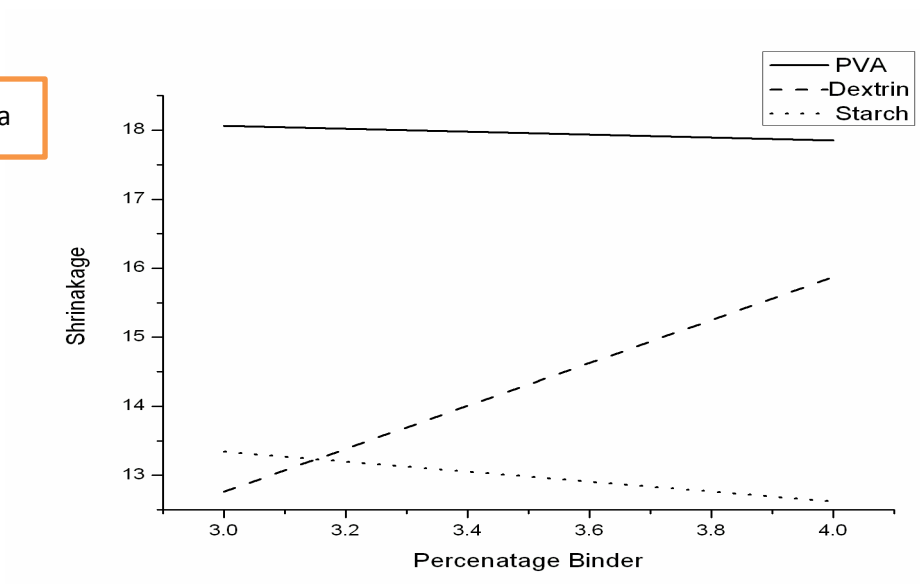

Fig. 12(a): Shrinkage VS Binder Percentage

b

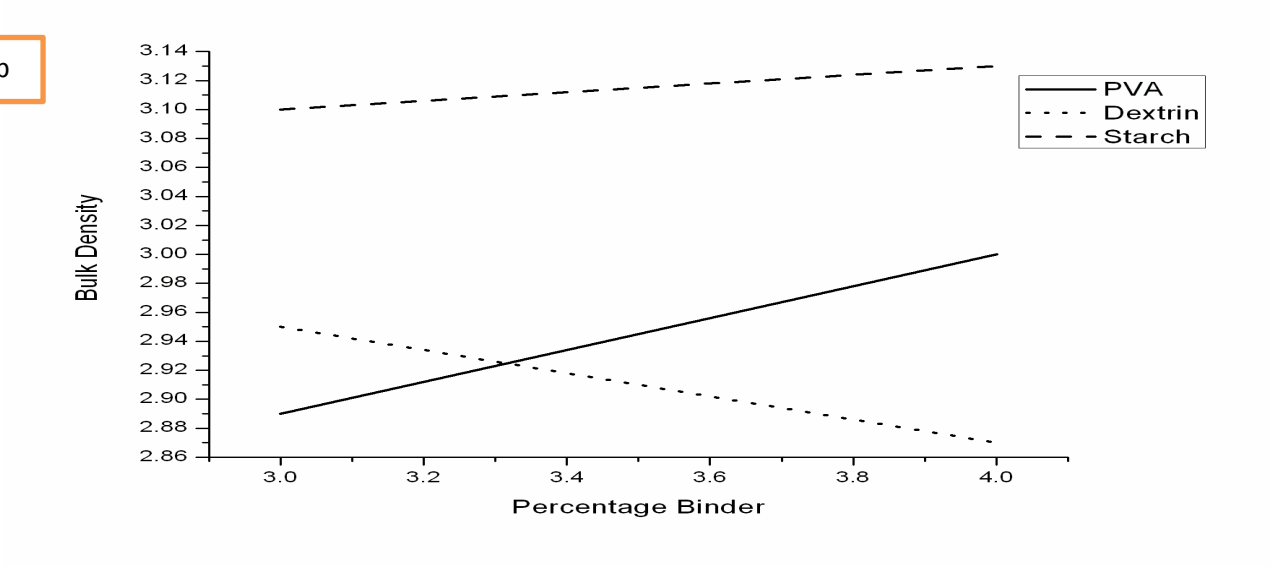

Fig. 12(b): Bulk Density VS Binder Percentage

c

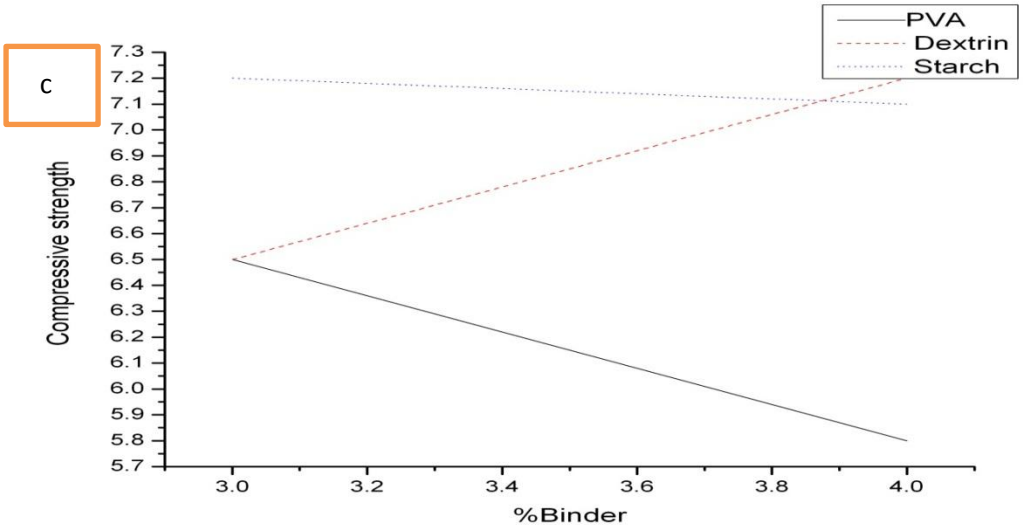

Fig.12 (c): Compressive strength (MPa) VS Binder Percentage

d

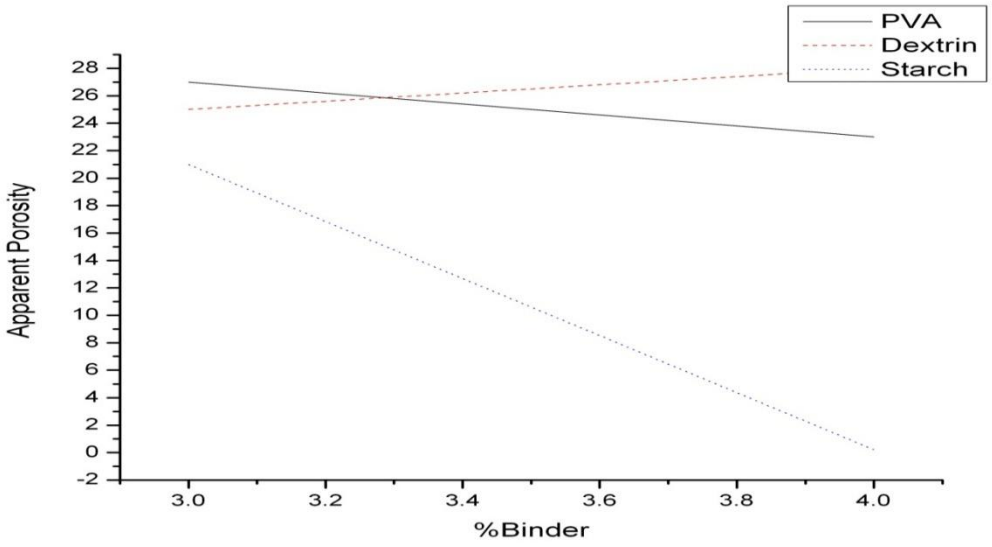

Fig. 12 (d): Apparent Porosity VS Binder Percentage

4.6 Batch 3 Analysis

Batch 3 samples were sintered at 2 ^{0}C/ minute up to 450 ^{0}C and the holding time at this temperature was 30 minutes. The results from the following graphs show that:

1. Shrinkage in PVA added samples is higher than other binder added samples

2. Bulk Density of Starch added samples is higher than that of PVA and Dextrin based samples.

3. The compressive strength of Starch (3 %) was the highest among the other binder added samples which may be due to the effective burnout at 450 ^{0}C.

Batch 4

a

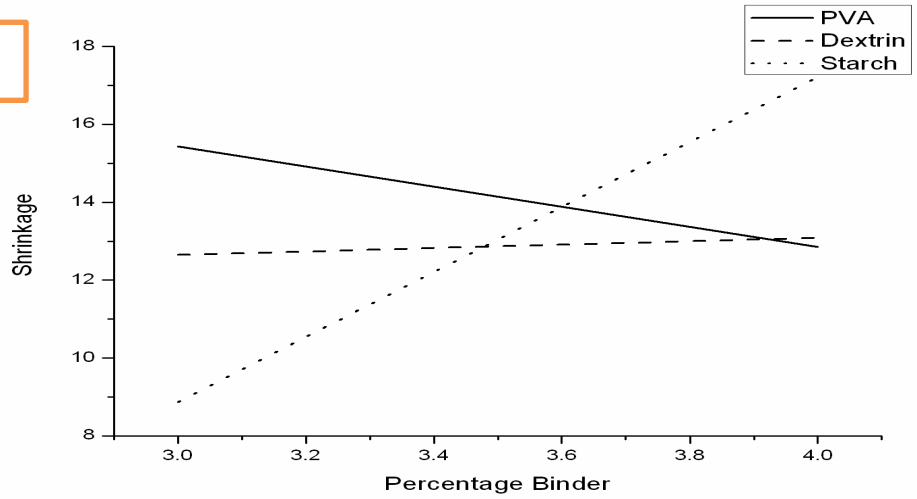

Fig. 13(a): Shrinkage VS Binder Percentage

b

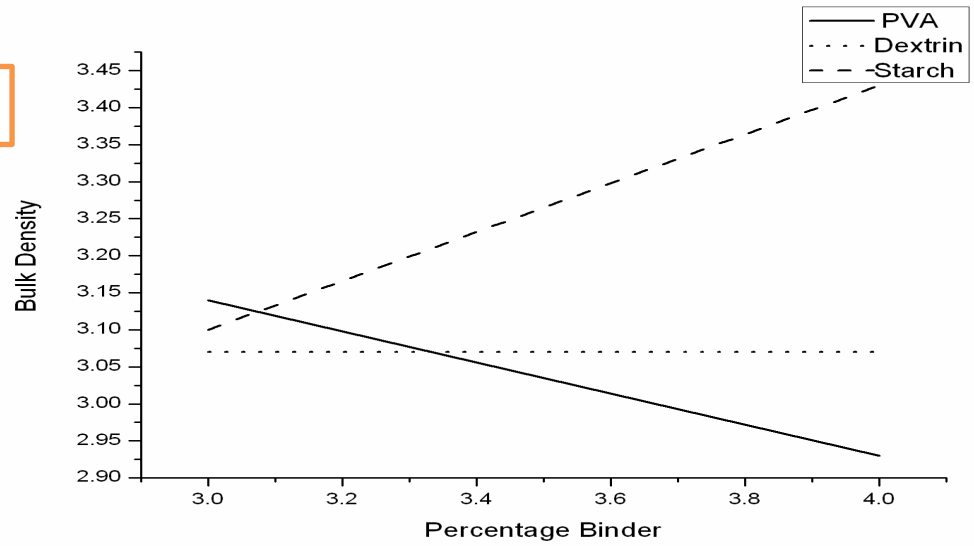

Fig 13(b): Bulk Density VS Binder Percentage

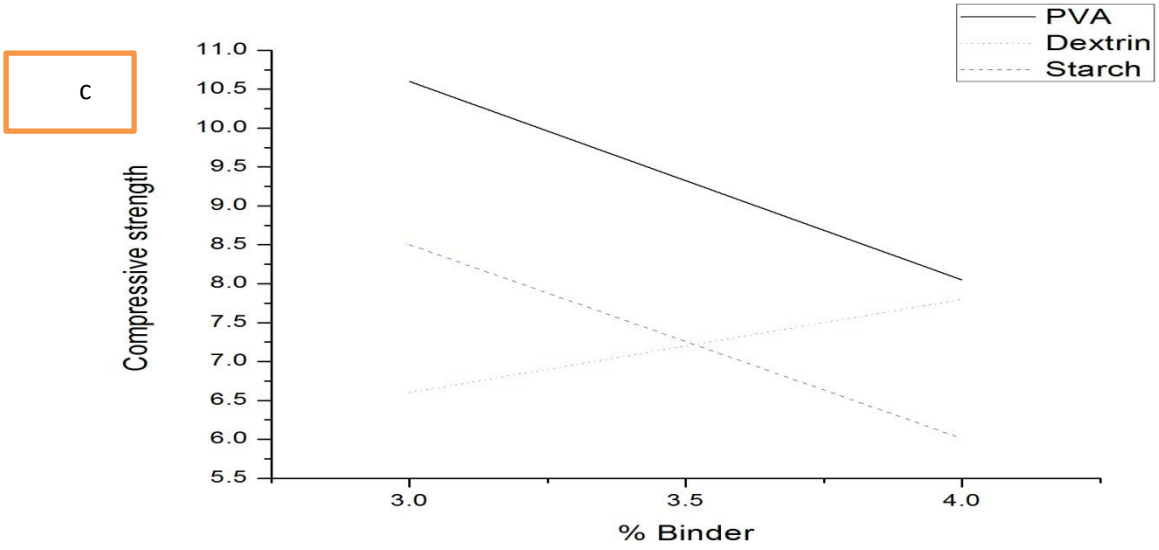

Fig. 13(c): Compressive Strength (MPa) VS Binder Percentage

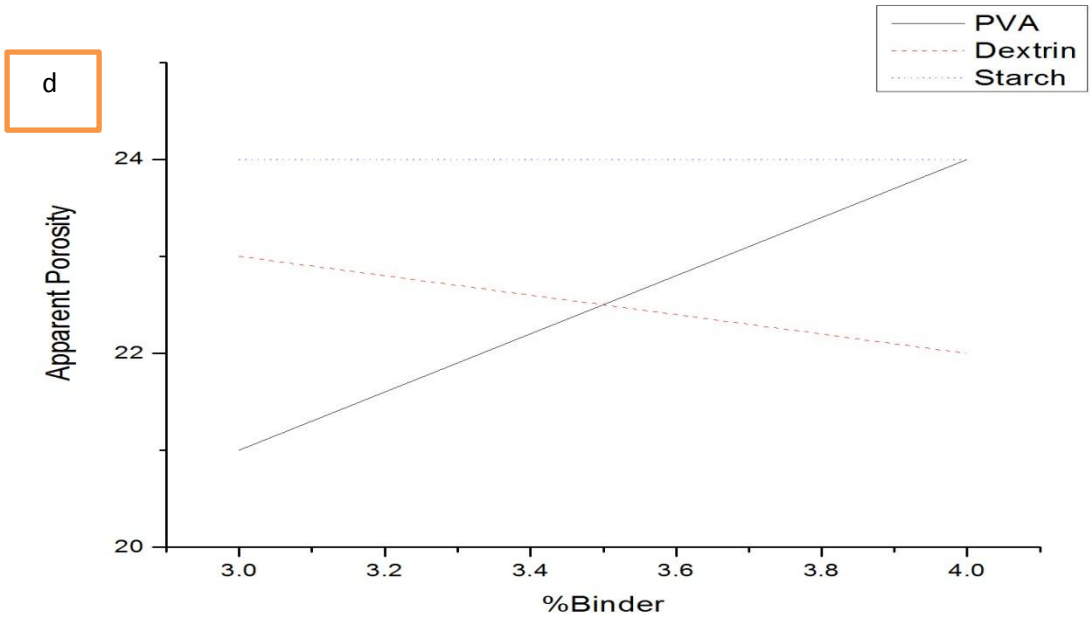

Fig.13 (d): Apparent Porosity VS Binder Percentage

4.7 Batch 4 Analysis

Batch 4 samples were heated at 450 ^{0}C at 2^{0}C/minute and the holding time was 60 minutes. The following conclusions can be drawn:

1. Starch added samples have higher bulk density than other binder added samples.

2. Starch (3%) based samples have higher compressive strength due to effective removal of binders.

3. PVA (3 %) based samples also had very high compressive strength. The increase in strength can be attributed to better burnout as the holding time was high for this batch.

CHAPTER 5

CONCLUSIONS

The conclusions derived from the project work are:

- Initially the project was to study the effect of both heating rate and holding time at binder removal temperature.
- However, due to furnace limitations, the project was modified to study only the effect of binder burnout with different holding time.
- Binder percentage has a marked effect on the green density, fired density and strength.
- The different binders used were starch, dextrin and PVA.
- In case of green bodies, PVA added samples showed highest strength.
- The mixture of PVA and reactive Alumina was viscous and this could be the reason for high strength.
- However, starch added samples showed minimum green strength.
- The mixture of starch and reactive Alumina was less viscous which might be the reason for the low strength.
- Starch added samples showed better strength under the performed heating schedule. Starch binder was better as it could go off before the sintering started and thus gave better strength and homogeneity to the sample.

Chapter 6

SCOPE FOR FURTHER WORK

1. The results from the data may be an overestimation of the strength of various binder added samples as the pellets under study were very small. Large size of pellets can significantly determine the effect of green density on the strength of the bodies. As the samples under study were small even a small change in density had a marked effect on the strength.

2. Few batches of samples showed high porosity in the range of 27 %. This can be reduced by firing the samples at temperatures higher than 1600 ^{0}C with greater holding time.

3. DSC-TG analysis of sintered pellets can give conclusive results of binder burnout in pellet atmosphere. DSC-TG analyses of binder powders have their limitations as the binder present in the pellets is not always homogeneously distributed across the system.

4. SEM analysis can be helpful in studying the microstructural properties of the sintered bodies and its relation with binder burnout and removal. Size of pores and pore size distribution can be determined to study binder burnout.

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