

GYPSUM PRODUCTS

Dept of prosthodontics, DYPDS pune

INTRODUCTION

- Gypsum is a naturally occurring mineral found in various parts of the world, particularly in Germany and Nova Scotia.
- Chemically, the gypsum is nearly pure *Calcium Sulfate Dihydrate* ($\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$).
- Gypsum was first found in mines around the city of Paris so it is called as *Plaster Of Paris*.



Gypsum under SEM

APPLICATIONS

A. GENERAL

For preparing statues and in construction work.

B. IN ORTHOPEDICS

For splinting and making plaster cast.

C. IN DENTISTRY

- Impression of oral and maxillofacial structures.
- To make moulds cast and dies over which dental prosthesis and restoration are made.
- To attach casts to an articulators.
- For bite registration .
- As dental investments.

CLASSIFICATION

According to ADA Specification No.25:

- Type 1 - Impression plaster.
- Type 2 - Dental plaster.
- Type 3 - Dental stone or medium strength stone.
- Type 4 - Improved stone or high strength stone.
- Type 5 - Dental stone, high strength, high expansion.

MANUFACTURE OF GYPSUM PRODUCTS

- The process of heating gypsum for manufacturing plaster is known as *Calcination*.
- Depending on the method of calcination, different types of gypsum products i.e calcium sulphate hemi hydrate are obtained.

MANUFACTURE OF GYPSUM PRODUCTS

110°-130°C

$\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$
Gypsum
(calcium sulfate dihydrate)



$\text{CaSO}_4 \cdot \frac{1}{2}\text{H}_2\text{O}$
Plaster or stone
(calcium sulfate hemihydrate)

130°-200°C



CaSO_4
Hexagonal anhydrite

200°-1000°C



CaSO_4
Orthorhombic anhydrite

CHEMICAL & PHYSICAL NATURE OF GYPSUM PRODUCTS

- Gypsum is the dihydrate form of CaSO_4 denoted as $\text{CaSO}_4 \cdot 2\text{H}_2\text{O}$.
- On heating it loses 1.5 g mol of its 2g mol of water & is converted to $\text{CaSO}_4 \cdot 1/2\text{H}_2\text{O}$
- When mixed with water , the reverse reaction is seen:



3900cal/gmol.

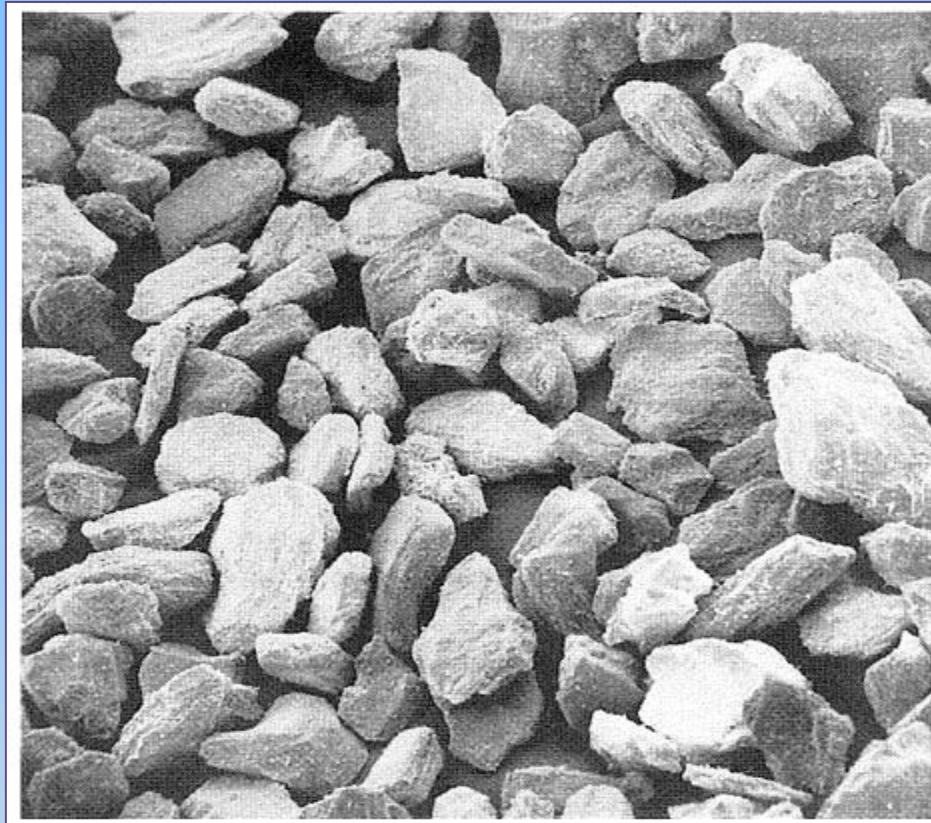
MANUFACTURE OF DENTAL PLASTER: (POP)

- Gypsum is heated to a temperature of about 110 to 120°C (230 to 250°F) in order to drive off part of the water of crystallization.
- This produces irregular, crystals with capillary pores- *β -hemihydrate*.
- Procedure is carried out in a kettle, vat or rotary kiln open to air –*Dry Calcination*.



MICROSCOPICALLY

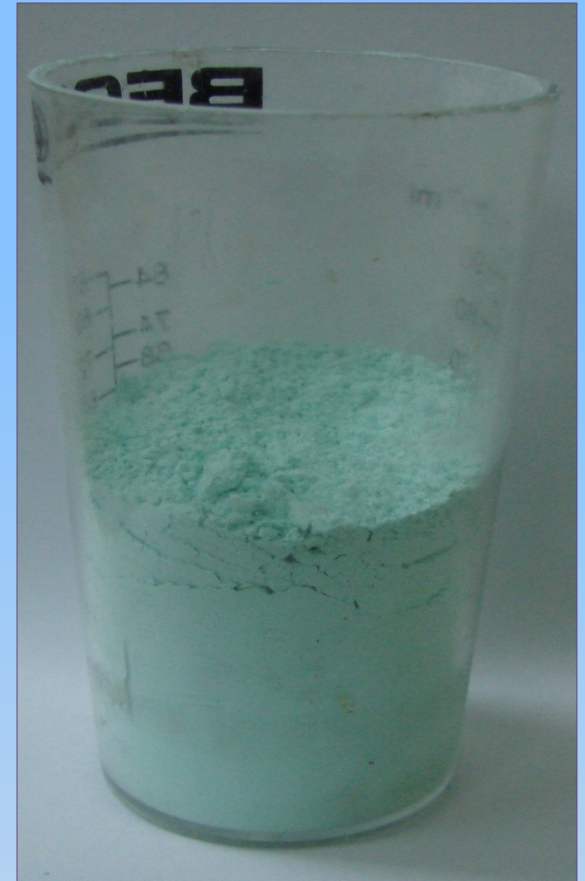
- Fibrous aggregate of fine crystals with capillary pores.



Powder particles of plaster of Paris (*β -hemihydrate*)

MANUFACTURE OF DENTAL STONE

- Gypsum is calcined under steam pressure in an autoclave at 120°C to 130°C at 17 lbs/sq. inch for 5 to 7 hours-*Wet Calcination*.
- Product obtained is much stronger and harder than beta hemihydrate.



MICROSCOPICALLY

- Crystals in the form of rods and prisms- **α hemihydrate**



Powder Particles of dental stone(α -hemihydrate)

Differences between α and β - hemihydrate

α -hemihydrate (Dental Stone)	β - hemihydrate (Dental Plaster)
Crystals in the form of rods or prisms and compact.	Spongy irregular in shape and porous.
Wet Calcination.	Dry Calcination.
Low setting expansion .	High setting expansion.
Set gypsum is stronger and harder.	Set gypsum is of lower strength.
High abrasion resistance.	Low abrasion resistance.

MANUFACTURE OF IMPROVED STONE

- Gypsum is boiled in a solution of a salt such as CaCl_2 (30%).
- CaCl_2 is washed away with hot water.
- They have even higher density & yield an even stronger set & are more resistant to abrasion.
- Microscopically cuboidal in shape.

THEORIES OF SETTING

- Colloidal theory / Gel theory
- Hydration theory
- Dissolution precipitation theory/ crystalline theory

WATER/POWDER RATIO

- **WATER/POWDER RATIO :**

Important factor in deciding the physical and chemical properties of the final product.

- ***Higher water-powder ratio:-***

Longer setting time & weaker will be the gypsum product.



WATER/POWDER RATIO AFFECTED BY:-

- Shape & compactness of crystals:-

Irregular, spongy plaster particles need more water than the denser stone.

- Small amounts of surface active materials like gum arabic plus lime(calcium carbonate) reduce water requirement of all gypsum products.
- Particle size distribution, grinding of the powder breaks up needle like crystals. Improves packing characteristics & reduces the water needed.

RECOMMENDED W/P RATIO

- * **Impression plaster : 0.50 to 0.75.**
- * **Dental plaster : 0.45 to 0.50.**
- * **Dental stone : 0.28 to 0.30.**
- * **Type IV : 0.22 to 0.24.**
- * **Type V : 0.18 to 0.22.**

EXCESS WATER

- The actual amount of water necessary to mix the calcium sulfate hemihydrate is greater than the amount required for the chemical reaction (18.61 gm of water per 100 gm of hemihydrate). This is called excess water.
- The excess water itself does not react with the hemihydrate crystals. It is lost by evaporation once the gypsum is set.
- The excess water serves only to aid in mixing the powder particles & is replaced by voids.

MANIPULATION:

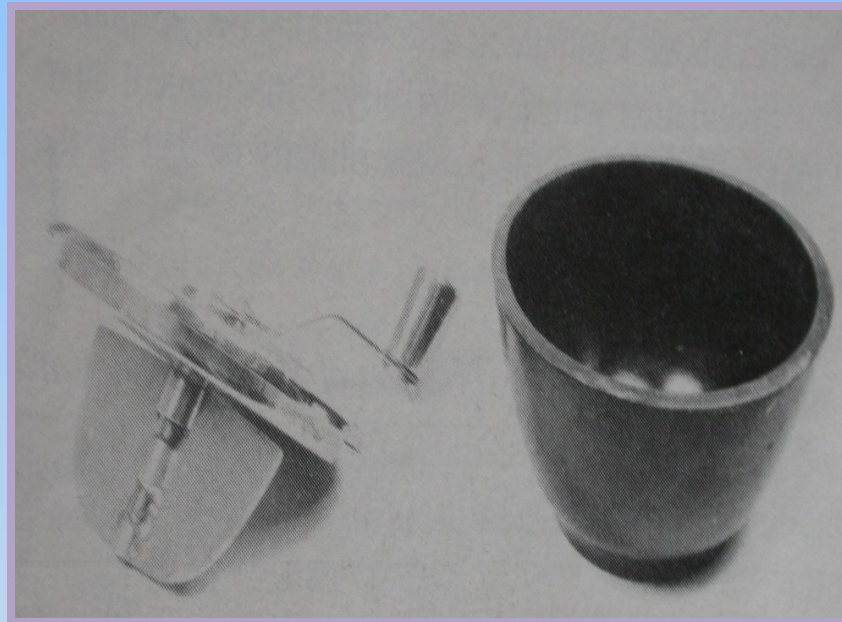
- A smooth mix should be obtained.
- Water is dispensed into a mixing bowl.
- The powder is added & allowed to settle into the water for about 30sec.
- It minimizes the amount of air incorporated into the mix.

**SPATULATION CAN BE CONTINUED BY HAND
USING A METAL SPATULA WITH A STIFF BLADE.**

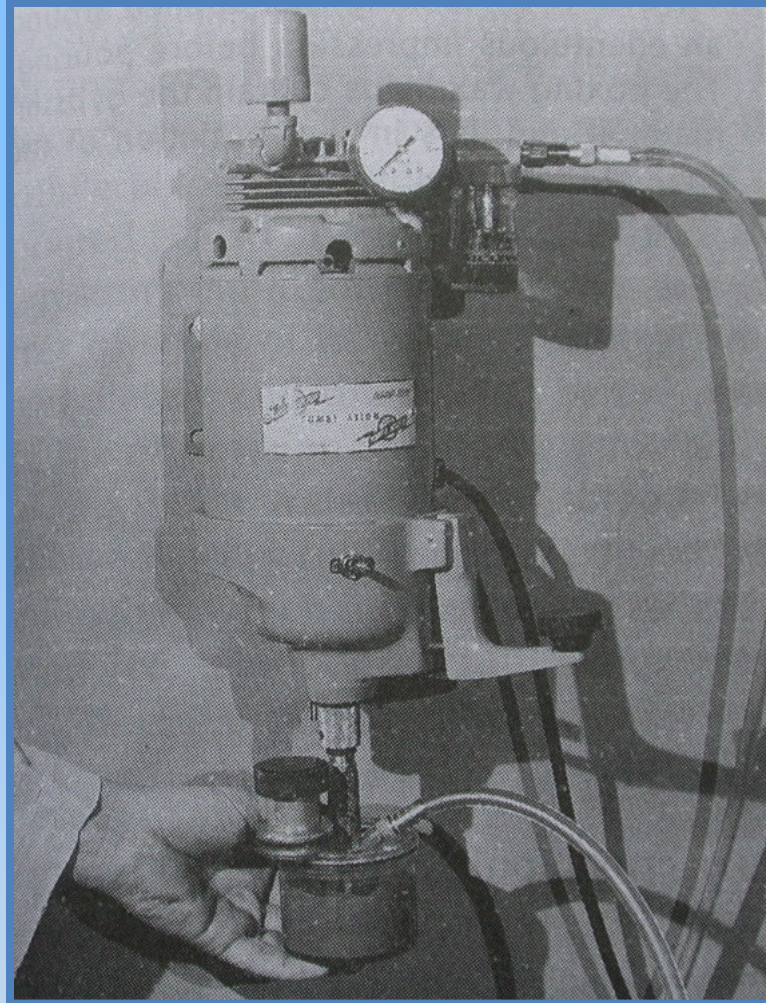


MECHANICAL SPATULATION

- Is to wet & mix the powder uniformly with the water required about 1min at 2 revolutions per sec.
- It involves stirring the mixture vigorously & at the same time wiping the inside surfaces of the bowl with the spatula.



A POWER DRIVEN MECHANICAL SPATULATOR.



PROPORTIONING

- Important to keep the amount of water as low as possible.
- Decreased amount of water increases the viscosity of the mix.
- The lowest limit of the viscosity permissible is governed by the ability of the water- plaster mixture to remain in position in the tray while the impression is being taken.

MIXING

- **BOWL:** flexible rubber/ plastic parabolic in shape.

It should be smooth & resistant to abrasion.

No corners in which powder can collect/ stagnate.

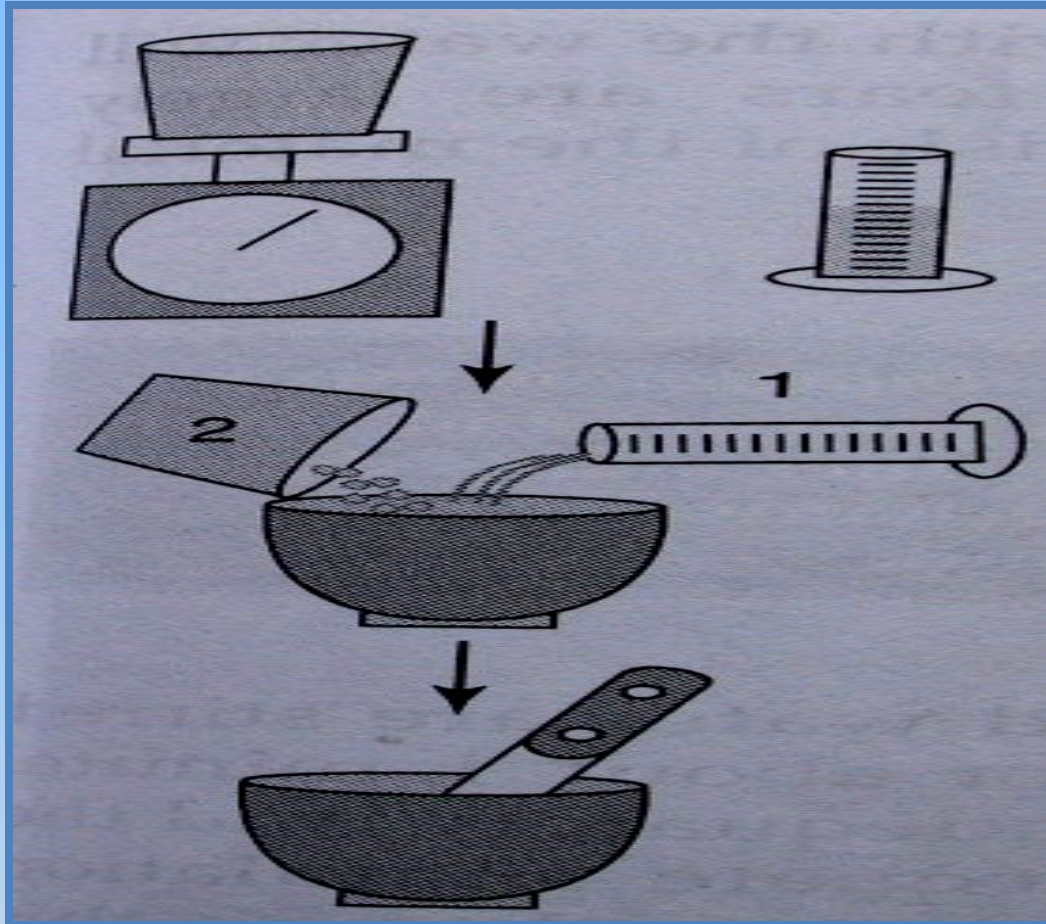
- **SPATULA:** stiff blade

A flexible blade drags when it is forced through a thick mixture.

End of the blade should be rounded to conform to the shape of the mixing bowl.

- **HANDLE:** readily grasped by hand.

MIXING



PROPERTIES

The important properties of gypsum products are :

- Setting time
- Setting expansion
- Strength
- Hardness and abrasion resistance
- Reproduction of detail .

SETTING TIME

MIXING TIME is the time from the addition the powder to the water until mixing is complete.

WORKING TIME is time available a workable mix ,i.e. one that maintains an even consistency that may be manipulated.

SETTING TIME The time elapsing from the beginning of mixing until the material hardens is called setting time.

INITIAL SETTING TIME :

As the reaction proceeds, however, more hemihydrate crystals react to form dihydrate crystals. The viscosity of the mass is increased, and it can no longer be poured. The material becomes rigid (but not hard).

It can be carved but not moulded. This is known as initial setting time.

FINAL SETTING TIME :

The time at which the material can be separated from the impression without distortion or fracture.

MEASUREMENT OF SETTING TIME

- **LOSS OF GLOSS METHOD**

As reaction proceeds, the gloss disappears from the surface of plaster mix (Sometimes used to indicate initial set).

- **EXOTHERMIC REACTION**

The temperature rise of the mass may also be used for measurement of setting time, as the setting reaction is exothermic.

- **PENETRATION TESTS**

By using penetrometers.

TYPES OF PENETROMETERS:

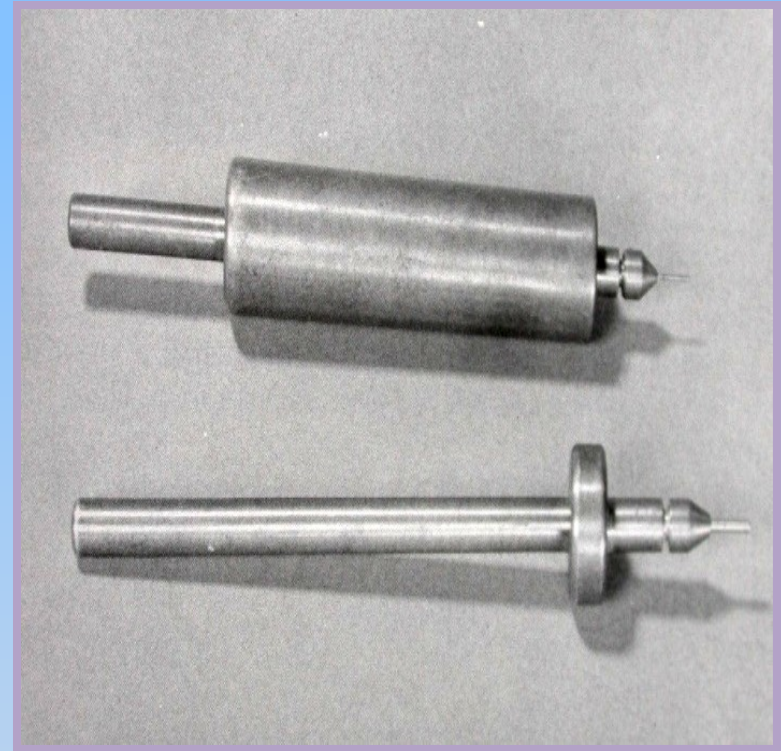
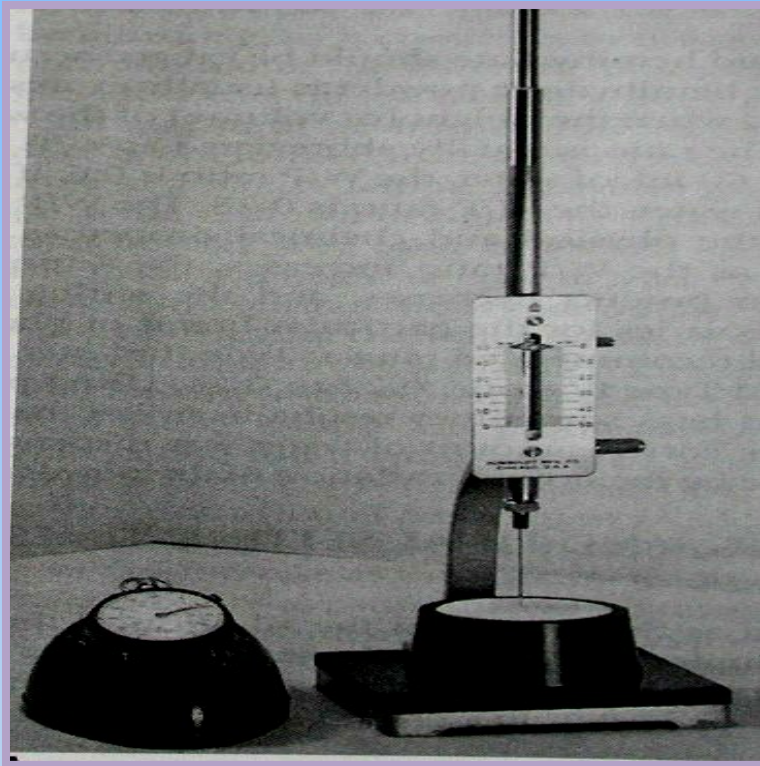
➤ **VICAT NEEDLE**

➤ **GILLMORE NEEDLE**

INITIAL GILLMORE TEST FOR INITIAL SET:

- It is measured in terms of their ability to resist penetration by needles.
- Heavier needles have a smaller tip diameter than lighter one and applies greater pressure

➤ The smaller 113.4 gms($1/4^{\text{th}}$ pound) needle has a 2.13mm point($1/12^{\text{th}}$ inch in diameter).



- The mixture is spread out and the needle is lowered on to the surface
- The time at which it no longer leaves an impression is called the initial set. Noted as ‘initial gillmore’.
- It indicates partial progress of a setting reaction.

GILLMORE TEST FOR FINAL SETTING TIME:

- The next stage in the setting process is measured by the use of the heavier gillmore needle - 453.6 gms(1pound) in weight and 1.06mm in diameter($1/24^{\text{th}}$ inch)
- The elapse time at which this needle leaves only a barely perceptible mark on the surface is called the *Final Setting Time*
- This is the time at which chemical reaction is practically completed

Ready for use criterion:

- It is a subjective measure of the time at which the set material may be safely handled.

VICAT TEST FOR SETTING TIME:

- The rod holding the needle weighs 300 gms, the needle is 1 mm in diameter and 5cm long
- Next stage in the reaction is determined by the *vicat penetrometer*
- The needle with a weighted plunger rod is supported and held just in contact with the mix
- The time elapse from the start of the mix until the needle no longer penetrates to the bottom of the mix is known as the setting time.

FACTORS AFFECTING THE SETTING TIME

- Manufacturing process.
- Mixing and spatulation time
- Water/ powder ratio
- Temperature
- Modifiers

FACTORS CAN BE DIVIDED INTO THOSE➔

■ CONTROLLED BY MANUFACTURER:

By control of nucleating agent in hemi-hydrate powder.

■ CONTROLLED BY OPERATOR:

Temperature

Water/ powder ratio

Mixing time

MANUFACTURING PROCESS

- **IMPURITIES**

When calcification is not complete so that gypsum particles remain, or if the manufacturer adds gypsum - Setting time shortened

- **FINENESS**

Finer the particles size of hemihydrate - the faster the mix hardens; and a more rapid rate of crystallization occurs.

▪ **MIXING AND SPATULATION**

Longer and faster the plaster is mixed -Faster it will set

▪ **WATER / POWDER RATIO**

More the water used for mixing ,the fewer the unit volume-Setting time will be prolonged .

▪ **TEMPERATURE**

If temperature of the mix increased beyond 50° C-retardation of setting time.

50° C- 100° C: converted back to hemihydrate.

100° C -No reaction take place.

▪ MODIFIERS (Accelerators and Retarders)

- ✓ Modifiers are chemicals added in order to alter some of the properties and make it more acceptable to the dentist.
- ✓ If the chemical added, it decreases the setting time, it is called an *Accelerator*
- ✓ If it increases the setting time ,it is called a *Retarder*.

ACCELERATORS

- Finely powdered gypsum (terra alba 1%)
- In low concentration –

Sodium sulfate (upto3.4%)

Potassium sulfate (2 to 3%)

Sodium chloride (upto2%)

RETARDERS

- Higher concentration – Sodium chloride and Sodium sulfate
(above 3.4%)
- Acetates
- Borates
- Citrates
- Tartates
- Inorganic salts like ferric sulfate, chromic sulfate, aluminum sulfate

RETARDERS

- Borax (1-2%) is the most effective retarder..

Colloids such as

- Gelatine
- Glue
- Agar
- Coagulated blood etc.

SETTING EXPANSION

- Normal setting expansion
- Hygroscopic setting expansion

NORMAL SETTING EXPANSION(0.05 to 0.5%)

All gypsum product show a linear expansion during setting, due to the outward thrust of the growing crystals during setting.

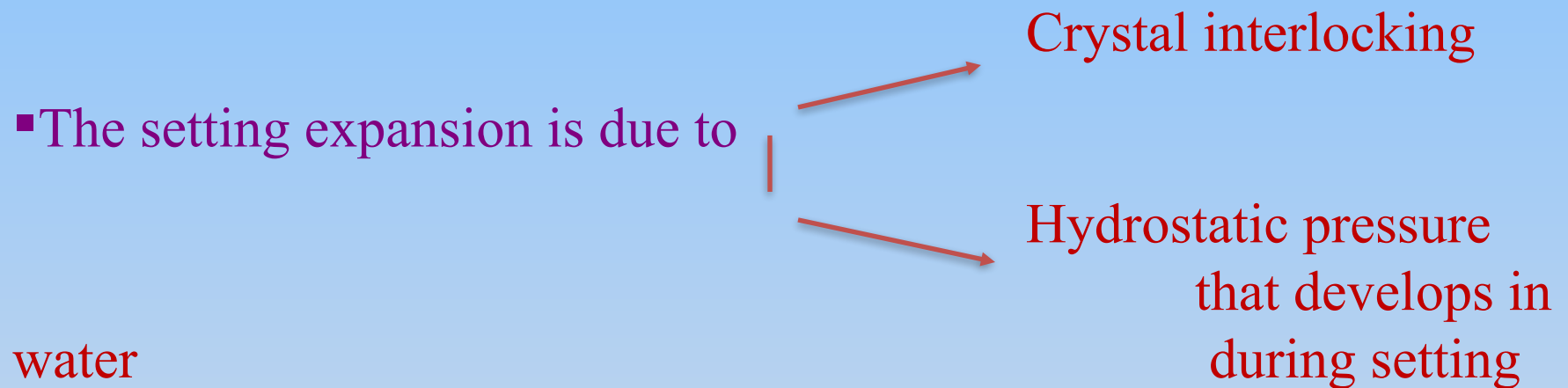


<u>MOLECULAR</u> <u>MASS</u>	290.284	54.048	344.332
<u>DENSITY</u> (gm/cm ₃)	2.75	0.997	2.32
<u>EQ. VOL.</u>	<i>105.556</i>	<i>54.211</i>	<i>148.405</i>
<u>TOT. VOL.</u>	159.767		148.405

■ THE NET CHANGE IN VOLUME IS -7.11%

▪ The setting expansion without water immersion is called *Normal Setting Expansion*

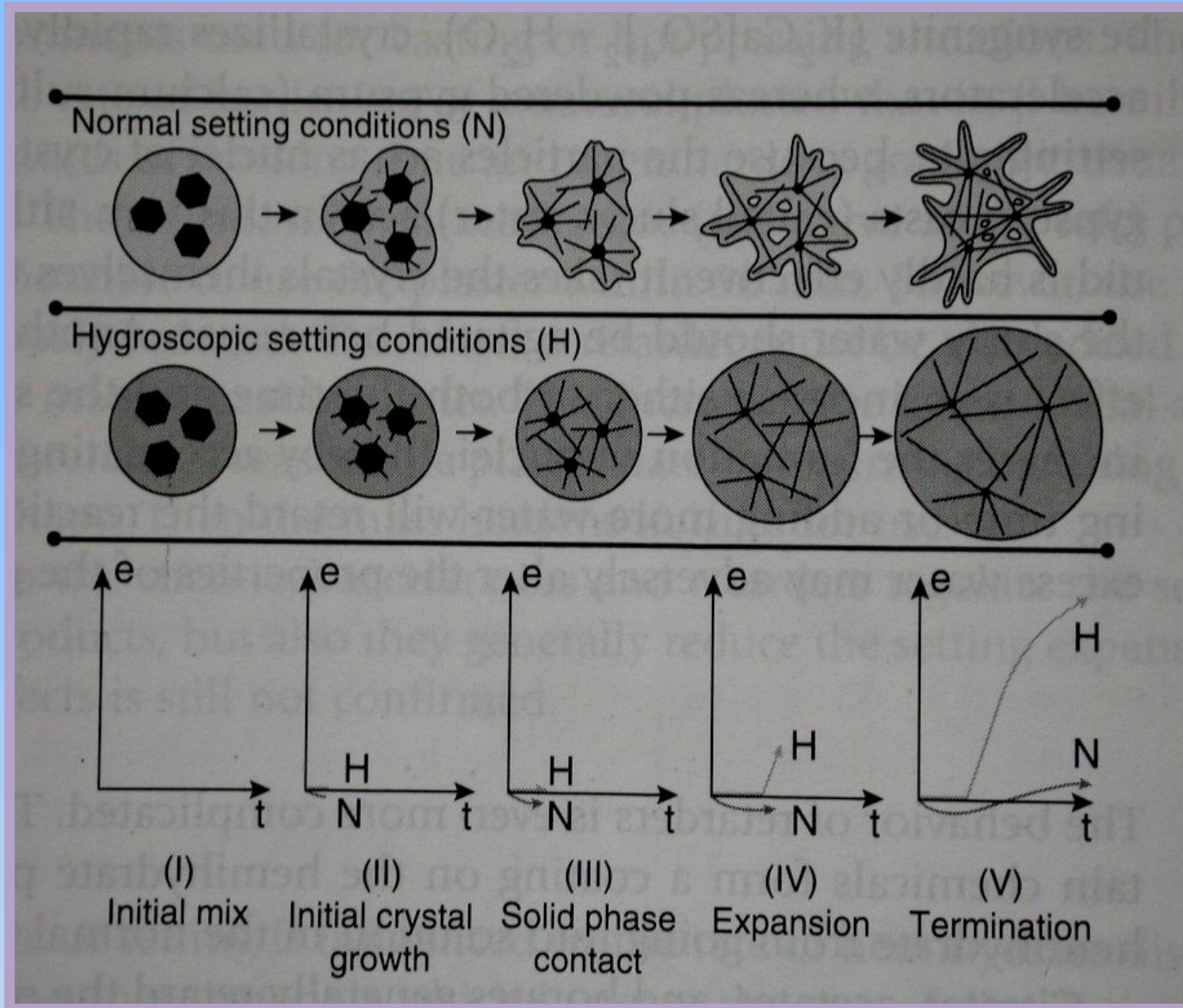
▪ Whereas that under water is known as *Hygroscopic Setting Expansion*



HYGROSCOPIC SETTING EXPANSION:

- If setting process is allowed to occur under water, the setting expansion may be more than double in magnitude.
- Reason for increased expansion is the additional crystal growth permitted by allowing crystals to grow freely, rather than being constrained by the surface tension when the crystals form in air
- The basic mechanism of crystal growth is the same in both.
- Both phenomenon are true setting expansion

HYGROSCOPIC SETTING EXPANSION



CONTROL OF SETTING EXPANSION

Increased Setting Expansion:

- Increased spatulation
- Sodium chloride and ground gypsum

Reduced Setting expansion

- Increase in W/P ratio.
- Modifiers-K₂SO₄.
- Sodium chloride
- Borax

IMPORTANCE OF SETTING EXPANSION

- Undesirable in impression plaster, dental plaster and stone as it will result in an inaccurate cast or change in the occlusal relation if used for mounting.
- Increased setting expansion is desired in case of investment materials as it helps to compensate the shrinkage of the metal during casting.

STRENGTH

- The strength increases rapidly as the material hardens after the initial setting time.
- Expressed in terms of Compressive strength
- The compressive strength is inversely related to the W/P ratio of the mix.

FACTORS AFFECTING STRENGTH

- **THE FREE WATER CONTENT;**

The greater the amount of free water in the set stone ,the less the strength

- **WET STRENGTH;**

Strength when the excess free water is present in the set gypsum.

- **DRY STRENGTH;**

Strength of gypsum when excess water is lost due to evaporation.

It is two or three time greater than the wet strength

- **TEMPERATURE:**

Gypsum is stable only below about 40°C.

Loss of water of crystallization occurs rapidly at 100°C - shrinkage and reduction in strength

- **W/P RATIO;**

The more the water ,the greater will be porosity and less the strength

- **SPATULATION;**

Within limits , the strength increases with increased spatulation

- **ADDITION OF ACCELERATORS AND RETARDER:**

Lowers strength

EFFECT OF W/P RATIO ON THE COMPRESSIVE STRENGTH OF GYPSUM PRODUCTS

MATERIAL	W/P RATIO	COMPRESSIVE STRENGTH (MPa)
Model plaster	0.45	12.5
	0.50	11.0
	0.55	9.0
Dental stone	0.27	31.0
	0.30	20.5
	0.50	10.5
Dental stone,(High strength)	0.24	38.0
	0.30	21.5
	0.50	10.5

TENSILE STRENGTH

- Gypsum is brittle material ;thus weaker in tension than compression.
- The one hour tensile strength of model is approximately 2.3 MPa .when dry tensile strength doubles .
- Tensile strength of dental stone is twice than that of plaster .

HARDNESS AND ABRASION RESISTANCE

- This is related to the compressive strength .
- The higher the compressive strength of the hardening mass, the higher the surface hardness.
- The hardness of gypsum product increased by impregnating the set gypsum with epoxy or methyl methacrylate monomer.
- Impregnating set gypsum with resin increases abrasion resistance.

REPRODUCTION OF DETAILS

- Gypsum dies do not reproduce surface details as well as electroformed dies or epoxy dies because surface of set gypsum is porous on a microscopic level .
- Air bubbles are often formed at the interface of impression and gypsum cast because freshly mixed gypsum does not wet some rubber impression materials well .

- The incorporation of some nonionic surfactant in polysulfide and silicone impression material improves the wetting of impression.
- The use of vibration during pouring reduces the presences of air bubbles. Gypsum products reproduce detail accurately.

TYPE 1 OR IMPRESSION PLASTER

- Impression plaster was one of the earliest impression materials in dentistry.
- Fractured to remove it from undercut areas in the mouth.
- The fractured pieces were then reassembled outside and a cast was poured.
- Plaster is primarily restricted to a final or wash impression in the construction of full denture.



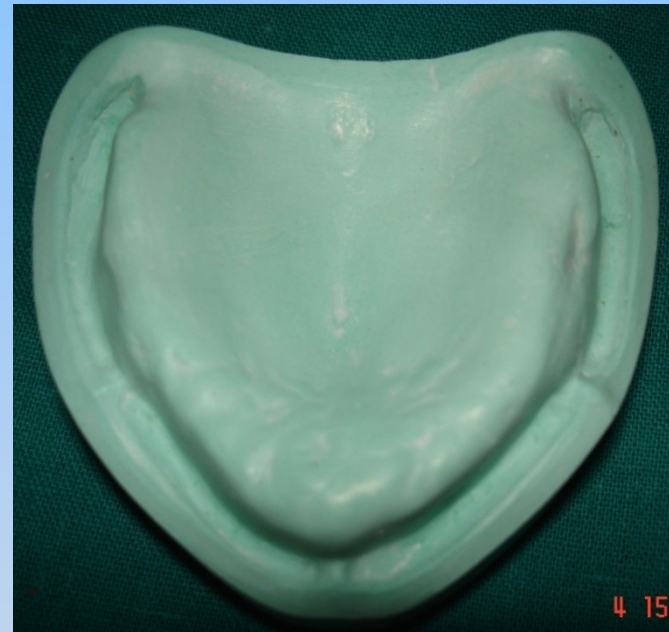
MODEL PLASTER (TYPE- II)

- This model plaster or laboratory type II plaster is now used primarily to fill a flask in denture construction when setting expansion is not critical and strength is adequate.



DENTAL STONE (TYPE III)

- It is intended for construction of cast in fabrication of full denture that fit soft tissues.



DENTAL STONE , HIGH STRENGTH TYPE(IV)

- Synonyms Die stone
- Die stone is the strongest and hardest variety of gypsum product. It is used when surface hardness and high strength is required ,e .g. dies used inlay ,crown and bridge wax patterns.



TYPE V OR DENTAL STONE ,HIGH STRENGTH, HIGH EXPANSION

- Higher compressive strength than type IV stone
- Improved strength is attained by making it possible to lower w/p ratio even further .
- Setting expansion is increased from a maximum of 0.1% to 0.3% This is for compensating for the shrinkage of base metal alloys ,during solidification.

TYPICAL PROPERTIES * OF THE FIVE TYPES OF GYPSUM PRODUCTS

Type	W/P ratio	Setting time (min)	2 – Hr setting expansion (%)		1- Hr compressive strength ⁺	
			Min	Max	(Mpa)	(psi)
1. plaster impression	0.40-0.75	4±1	0.00	0.15	4.0	580
II. Plaster, model	0.45-0.50	12±4	0.00	0.30	9.0	1300
III. Dental Stone⁺	0.28-0.30	12±4	0.00	0.20	20.7	3000
IV Dental stone, high strength	0.22-0.24	12±4	0.00	0.10	34.5	5000
V dental stone, high strength, high expansion	0.18-0.22	12±4	0.10	0.30	48.3	7000

SYNTHETIC GYPSUM:

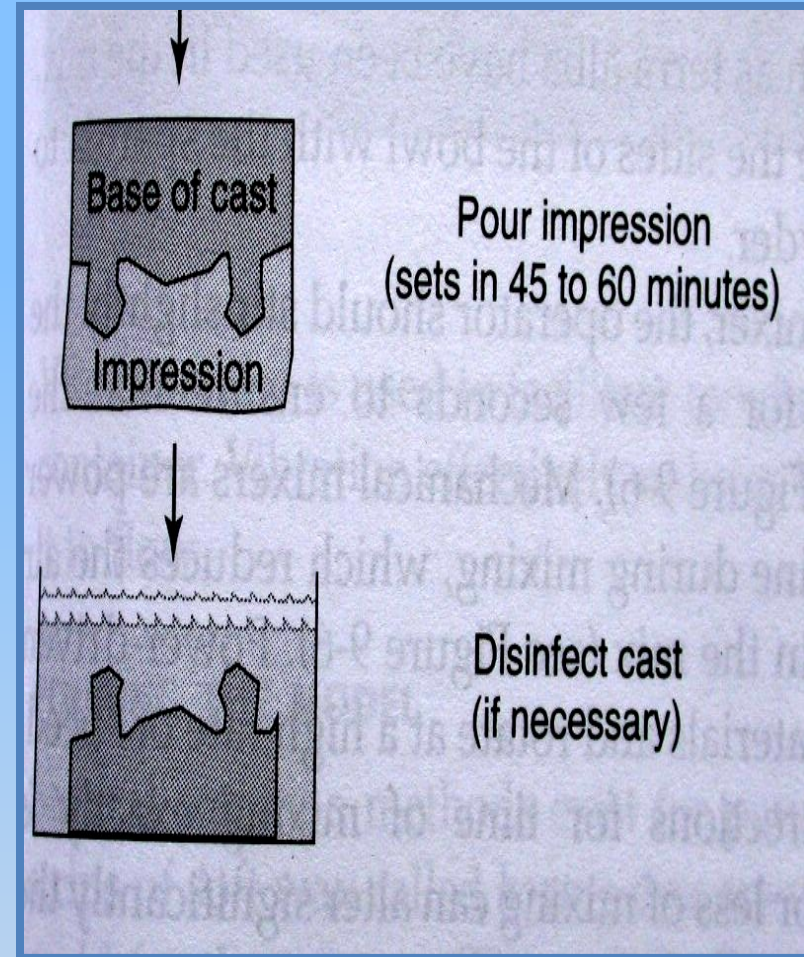
- Alpha & beta-hemihydrate are made from the byproducts or waste products of phosphoric acid production.
- Japan & Germany have been succeeded in its production.
- More expensive.

CARE FOR THE CAST

- Gypsum cast has to be soaked in '*slurry water*'.
- If the cast is washed in ordinary water, surface layer may dissolve, hence slurry water is used to preserve surface details. Such a procedure also causes a negligible expansion.

INFECTION CONTROL:

- 1) Iodophore sprays.
- 2) Immersion in disinfectant.
- 3) Overnight gas sterilization for patients with known cases.
- 4) 1:10 dilution of a 5% sodium hypochlorite for 30 min.
- 5) Ethylene oxide.



SUMMARY

A unique dental material which is easy to manipulate and sets to a hard and strong mass, because of its property it is widely used in dentistry, medical and for general purposes such as construction and statues, and other routine uses etc. Gypsum products are immensely useful in dentistry in general and prosthodontics in particular.

REFERENCES

- Dental Material's, Properties & Dental Manipulation
By R.G.Craig, O'brien, Powers
- Clinical Restorative Material's & Technique
By Leinfelder, Lemons
- Applied Dental Material's
By J.Mc CABE
- Skinners Science Of Dental Materials
By Ralph.W.Phillips
- The Clinical Handling Of Dental Material
By B.G.Smith & P.S.Wright

THANK YOU