Bharathidasan University

Tiruchirappalli – 620 024, Tamil Nadu, India

Programme: M. Sc. Physics

Course Title : Crystal Growth Methods and Characterization

Code : 22PH443

Unit II : Gel Growth

Dr. R. Ramesh Babu

Professor

Department of Physics

Gel Growth

Introduction

- Crystals grown in room temperature have relatively **less structural imperfection**.
- Gel method - room temperature which **yield relatively perfect crystals** for investigations.
- Gel technique becomes an elegant tool for studying growth - kinetics and mechanism of crystallization. It also helps to understand the influence of various parameters such as **temperature, gel pH, gel density, gel ageing, gel quality, nature and strength of the acid on the crystallization**.

WHAT IS GEL?

- A highly viscous two-component system of a semisolid nature, rich in liquid, and having fine pores in it may be referred to as ‘gel’.

- Silica gel, known as silica hydrogel (or waterglass, or silicate glass) is usually prepared from Sodium Meta Silicate Solution (SMS).

- Agar gel (a carbohydrate polymer derived from seaweeds),

- gelatin gel (which resembles protein structure),

- clay gel, soap fluid, polyacrylamide, dense solution of metal hydroxides, polyvinyl alcohol,, aluminates, etc.

WHAT IS GEL?

It may be mentioned that **silica hydro gel** has been most commonly used, due to its better suitability than all organic gels.

However, in certain specific cases **gelatin gel, agar gel and polyacrylamide** have been preferred;

In some cases both the **inorganic and organic gels** have been found equally good for crystal growth .

GELLING MECHANISM AND STRUCTURE OF SILICA GEL

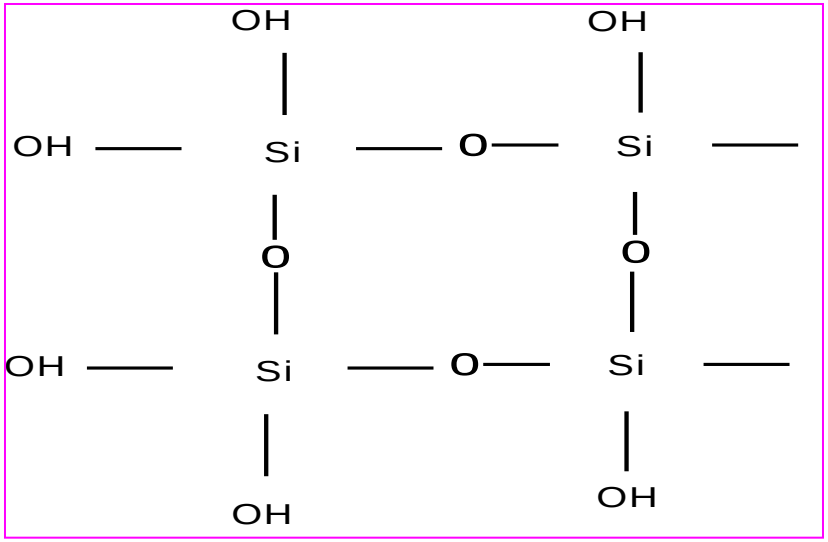
When the sodium meta silicate is mixed with water, monosilicic acid is produced which is represented as

$$Na_2SiO_3 + 3H_2O \rightleftharpoons H_4SiO_4 + 2NaOH$$

One of the interesting properties of monosilicic acid is that it polymerizes by liberating water.

GELLING MECHANISM AND STRUCTURE OF SILICA GEL

This process repeats and a three dimensional network of Si-O links, as shown below, is established



Preparation of gel solution

The Sodium Meta - Silicate (SMS) solution can be prepared by taking commercially available (water glass) the sodium meta silicate and double distilled water in the ratio 1:2 in a container and it is to be shacked well.

The container is then left undisturbed for about fifteen days to allow for sedimentation.

The clear solution is then filtered and stored in a separate well cleaned container. This solution is known as stock solution and density of the stock solution is measured (about 1.2 g/cc).

A survey of literature reveals that the suitable gel density for growing crystals is in the range of 1.02 – 1.05 g/cc.

GROWTH PROCEDURE

To all outward appearances, growing crystals in gel is exceedingly simple. But actually the physical and chemical processes, which determine its outcome, are not simple. The basic principle underlying all methods of growing crystals in gels is as follows.

Aqueous solutions of two suitable compounds which give rise to the required insoluble substance is allowed to react in a gel medium.

AX and BY be the two water soluble compounds which on reaction produce the required insoluble substance AB and an unwanted product XY soluble in water.

$$AX + BY \longrightarrow AB + XY$$

GROWTH PROCEDURE

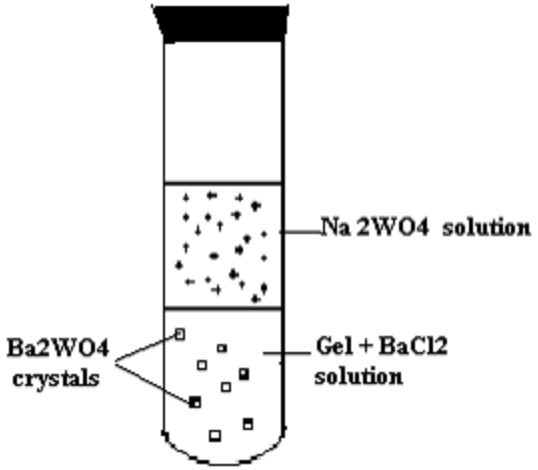
The gel technique can be categorized into the following four methods:

- Reaction method
- Chemical reduction method
- Complex decomplexion method
- Solubility reduction method

Growth of $BaWO_4$ crystal by reaction method

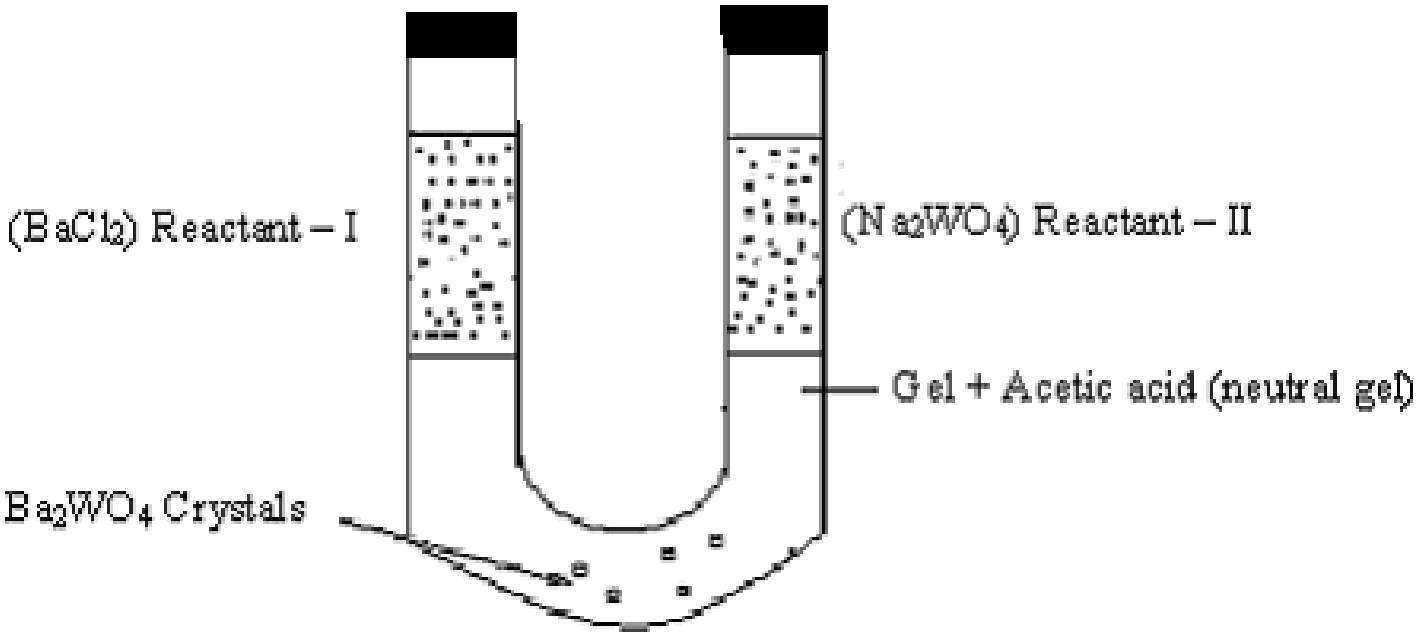
$$BaCl_2 + Na_2WO_4 \rightarrow Ba_2WO_4 + 2NaCl$$

Ba_2WO_4 crystals are insoluble in water and NaCl is soluble in water.



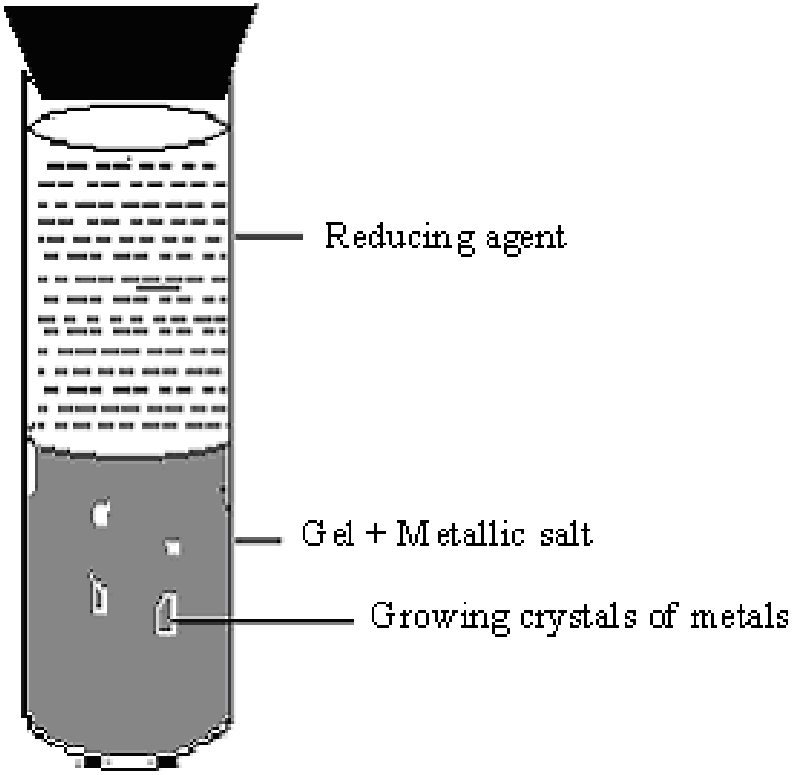
Single diffusion method

Growth of $BaWO_4$ crystal by reaction method

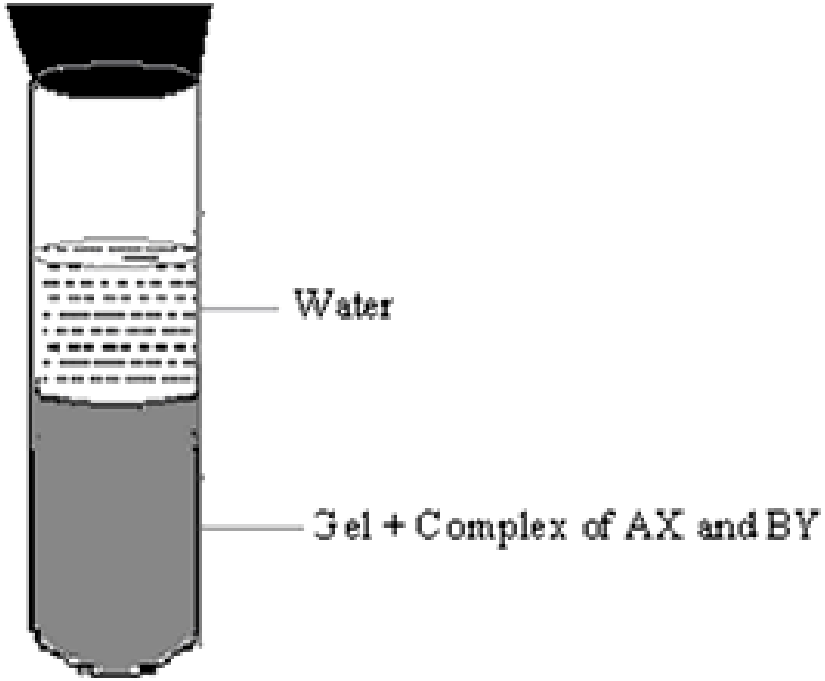


Double diffusion method

Chemical Reduction Method

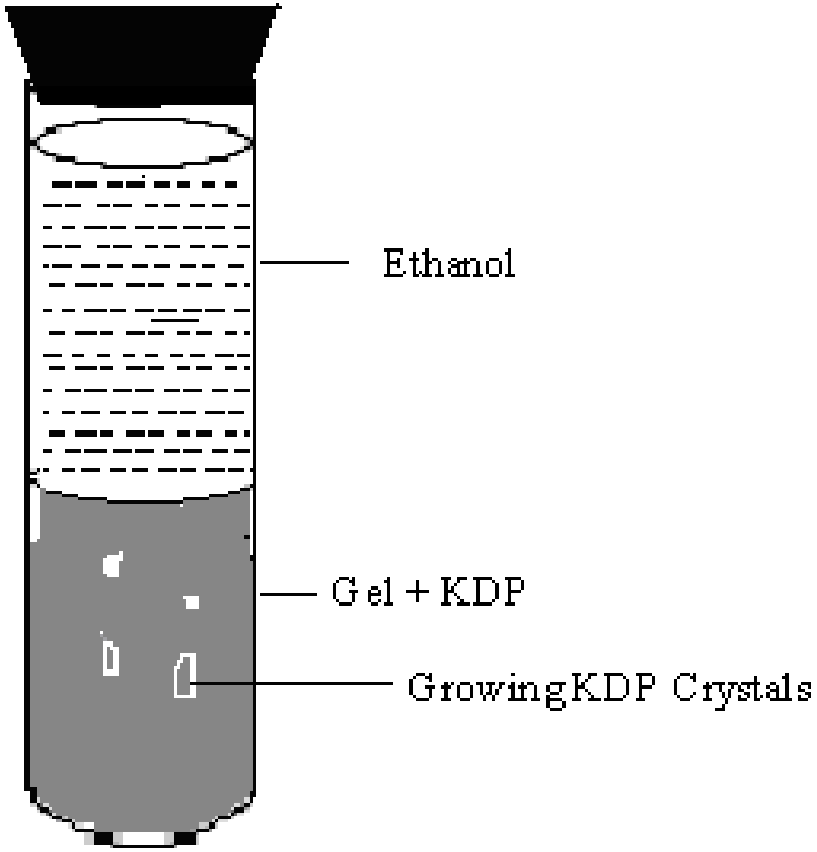


Complex Decomplexion Method



Complex dilution method

Solubility Reduction Method



References

H. K. Henisch, in *"Crystal growth in Gels and Liesegang Rings "*, Cambridge University Press, Cambridge, (1988)

S. K. Arora Prog. *Crystal Growth Charact*. 4 (1981) 345.

H. K. Henisch, in "*Crystal Growth in Gels*", The Pennsylvania State University Press, University Park, London (1973)

P. Santhana Raghavan and P. Ramasamy, *Crystal Growth Processes and Methods*, KRU Publications, Kumbakonam (2001).