

PREPARATION OF COLLOIDAL BISMUTH HYDROXIDE

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Introduction

Bismuth compounds were first tried in the end of 18th century for treating syphilis and gonorrhoea. Robert and Sauton¹ discovered the bactericidal properties of bismuth compounds on *Spirochaeta gallinarum* which paved the way for the treatment of syphilis. Until then this disease was being treated solely with arsenic preparations. Bismuth compounds have now become an adjunct to arsenicals in syphilis therapy. Hydroxy salts of bismuth being astringents serve as local antiseptics likewise bismuth subgallate in eczema is used as a dusting powder. Bismuth subcarbonate is employed in gastritis and enteritis. Oily suspension of bismuth salicylate was used in syphilis before the advent of penicillin.² Colloidal bismuth hydroxide was also employed for the treatment of syphilis³ and diarrhea.⁴ Bismuth also has a specific action on styphylcoccal infection.⁵

The first preparation of colloidal $\text{Bi}(\text{OH})_3$ was reported by W. Blitz⁶ in 1902 who dialysed bismuth nitrate solution in dilute HNO_3 . K.C. Sen and N.R. Dhar⁷ made colloidal $\text{Bi}(\text{OH})_3$, in 1923, with a view to study its peptization behaviour and reversal charge on metal hydroxides by agents like, glycerol, sucrose, fructose, mannitol and starch. 1.5 c.c. of 0.1N NaOH was added to 1 c.c. of 0.1N salt solution containing different peptizing agents. In 1925 A. Khun and H. Pirsch⁸ prepared electrolyte free colloidal $\text{Bi}(\text{OH})_3$ both by centrifuging and by dialysing. They added substances like raw sugar and glycerol to stabilize the peptized hydroxide. C. Paal and Leonardo di Pol⁹ made it in 1926, by the reduction of $\text{BiO}(\text{OH})$ solution with, a solution of formaldehyde and NaOH, 30% and 2 N., respectively by refluxing for a long time and cooling in an atmosphere of CO_2 . The above work was undertaken to study the peptization behaviour of metal hydroxides towards the electrical conductivity of alkali solution which is not appreciably changed. For pharmacological studies Alfredo Chistoni,¹⁰ in 1925, prepared

colloidal $\text{Bi}(\text{OH})_3$ by heating bismuth phosphate with NaOH in the presence of albumin.

Keeping in view the therapeutic importance of bismuth hydroxide the authors prepared its colloidal solution with sucrose in the presence of NaOH. This preparation takes less time in production and is electrolyte free.

Preparation

2.321 g. of bismuth nitrate, $\text{Bi}(\text{NO}_3)_3 \cdot 5\text{H}_2\text{O}$, (B.D.H. quality,) were dissolved in 50 ml. of conc. HNO_3 and diluted with 40 ml. of distilled water. Bismuth hydroxide was precipitated by adding 25 ml. of 15% NaOH at room temperature. This hydroxide was washed six times with 450 ml. distilled water to remove electrolytes, decanting each time before the addition of water. Freshly prepared bismuth hydroxide was used as the starting material in the preparation of its colloidal form. The quantity of hydroxide taken was equivalent to 1 g. of elemental bismuth, and was mixed with 20 g. of sucrose in 25 ml. distilled water to which 35 ml. of NaOH were added and the mixture heated to 70°C. until it became clear. The function of sucrose is to stabilize the colloidal bismuth hydroxide solution. The same function has been observed¹¹ with the preparation of colloidal iron hydroxide. The solution gave a pH of 12.5 and did not react with KI and thiourea. On passing H_2S it gave a black precipitate of Bi_2S_3 . The isoelectric point was determined with N/10 HCl and was found to be pH. 2.0. Ampules of 1% solution were autoclaved for half an hour. The solution remained stable thus confirming its colloidal state. On autoclaving further for an hour, it gave a black precipitate, which indicated reduction of the hydroxide into its oxide. On heating for one hour, upto 125°C., on direct flame, it gives a brown precipitate, but this, with further addition of water in excess and keeping for 24 hours, again goes into solution only with slight turbidity. Alcohol precipitates the colloidal bismuth sucrose solution. The precipitate is whitish in colour and crystalline in nature which is being further studied. Estimation of bismuth was done colorimetrically by developing colour with thiourea and calculating in percentage.

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to become separated so that their distinguishing characters can be studied thoroughly.

Different workers have followed different methods of maceration of the fibre cells. It was thought earlier that water soluble salts of sodium, potassium or ammonium with any acid (organic or inorganic) whose calcium and magnesium salts are insoluble or practically so, would be good retting agents. Ordinarily 1% solution of the salts at 70°-80°C. for 1-4 hours is sufficient for satisfactory retting.¹ Johnson² used Jefferey's method according to which the materials were macerated in a solution of equal parts of 10% aqueous Nitric acid and 10% aqueous Chromic acid. In his opinion this was the simplest method as compared with the one proposed by Harlow³ and others. However, the case of breakdown of plant tissues is influenced by the degree of hydrolysis of pectic cell walls, rate of diffusion of enzymes and concentrations of enzyme extract.⁴

The methods used by these workers have not been found very suitable for proper retting of the materials and consume more time. Moreover, the material is either broken or becomes curved and remains thick due to the presence of gummy and pectic substances, thus making the study difficult. The author, therefore, has developed an improved method which overcomes the above difficulties.

Procedure

The strips of the bark are collected from the internodes of the main stem and fixed in Formalin-acetic alcohol solution (Formalin-10 c.c., glacial acetic acid-5 c.c. and 70% alcohol-85 c.c.). It can also be kept in distilled water for a day or two.

While studying the fixed material, it should be washed first with water. The bark is scrapped with scalpel to remove the periderm. The thin layer is taken out from outer, middle or inner surface as is required. The material is then boiled in retting solution comprising 1 gm. of caustic soda, 2 gm. of ammonium oxalate, 4 gm. of sodium sulphite, 10 drops of nitric (conc.) acid in 100 c.c. of distilled water for 15 minutes (10 minutes for extracted dry fibre). After boiling, it is washed with cold water, cleaned in a watch glass and placed on a slide for final scrapping. The material is again boiled in retting solution for 15 minutes. After washing and cleaning it again, the material is boiled with distilled water for 10 minutes (5 minutes for extracted dry fibre). It is then boiled in 5% chromic acid for 3-4 minutes and washed thoroughly twice or thrice in a watch glass. The material is now ready for staining and examination.

A NEW TECHNIQUE FOR MACERATION OF FIBRE TISSUES

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Commercially, fibre is generally, extracted by biological process of retting. But in a laboratory, different chemical processes have been employed for the extraction of fibre to ascertain their quality and other characteristics.

However, to study the individual fibre cells, the retted material is macerated so that the real nature or structure of the individual cell is exposed. This is a process through which bark of the stem or other parts of the plant is treated with chemicals dissolving the middle lamellae and other pectic and gummy substances allowing the cells

For microscopic study, the material is taken on a watch glass with 1% methylene blue and washed with distilled water to remove the over-staining. A drop of 20% glycerine is placed on the slide along with a small piece of the material. Single strands of fibre are separated under a dissecting microscope with a sharp pointed needle, a cover slip is placed on the slide and examined under the microscope.

The present method is a modification of the methods developed by earlier workers, and is being used in this Laboratory. This method is found to be advantageous over other methods because the gummy and other pectic substances are completely separated from the ultimate fibres, thus facilitating the study of the latter.

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