Solvothermal and Hydrothermal Techniques for Nano-synthesis: An Overview

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Preface

Nanotechnology has emerged as a transformative field, reshaping industries, from healthcare to materials science, by manipulating matter at the nanoscale. Hydrothermal and solvothermal techniques are cornerstones of nano-synthesis, offering versatile and efficient methods for crafting nanomaterials with precision. These methods have played an integral role in harnessing the unique properties of nanomaterials, enabling advancements in electronics, catalysis, energy storage, and countless other domains.

The inspiration behind this booklet is to provide a comprehensive introduction to hydrothermal and solvothermal techniques, making this complex field accessible to both beginners and seasoned researchers. Whether you're a scientist seeking to expand your knowledge or an enthusiast eager to explore the wonders of the nanoworld, this booklet is designed to be your companion. In this booklet, "Hydrothermal and solvothermal techniques for nano-synthesis: An overview" by Dr. Manisha Bora, you will find an exploration of the fundamentals of hydrothermal and solvothermal synthesis, the intricacies of reaction parameters, and the diverse applications of nanomaterials that arise from these techniques. I congratulate Dr. Manisha for writing this booklet.

We must recognize that nanoscience is not just about shrinking materials but expanding our horizons. The nanoworld presents us with an array of challenges and opportunities, and the techniques explored within this booklet are our tools for navigating this frontier.We hope this booklet serves as a valuable resource, inspiring curiosity and innovation in the realm of nanosynthesis.

Whether you are an academic, a researcher, or simply someone intrigued by the endless possibilities unlocked by nanotechnology, we invite you to join us on this exploration of hydrothermal and solvothermal techniques for nano-synthesis.

On behalf of ISAS, Pune Chapter I would like to thank ISAS President Dr. Raghaw Saran for his encouragement in implementing booklets project. We are thankful to EC members of ISAS, Pune Chapter and booklet scrutiny committee for their approval in undertaking this task. I am thankful to Dr. Vinay Bhandari, Dr. Narendra Gokarn, Sanjay Bhandare and Santosh Khutwad for their cooperation. My sincere thanks are due to Dr. Manisha Bora for Financial Assistance towards booklet printing.

Dr. Nilima Rajurkar
Chairperson
ISAS, Pune Chapter

Preface

I am glad to introduce solvothermal and hydrothermal techniques for the synthesis of nano-materials. This is one of the several techniques which are very popular among material scientists as they are eco-friendly, economical and may be used for preparing nanomaterials in high quantity. The various materials prepared by these methods provide unique morphologies with narrow range of particle size. The hydrothermally synthesized materials have been exploited for numerous applications by researchers all over the globe.

I am deeply obliged to the Chairperson, ISAS Pune chapter Dr. Nilima Rajurkar, for her immense support, guidance, encouragement, valuable suggestions and tireless efforts towards the publication of this booklet in time. I also than to Dr. Narendra Gokarn, Member, ISAS Pune Chapter for proof reading the manuscript and giving his valuable suggestions. I thank Dr. Nilima Rajurkar, Secretary Dr. Avinash Kumbhar, and Treasurer Dr. Sunil Hande, Joint secretary Dr. Anupa Kumbhar for providing such wonderful platform, which provides academicians like me to nurture my research competencies, writing skills and many other skills. I extent my sincere thanks to thank publishers, for timely printing and publishing this booklet. I also thank management, Principal Dr. Sanjay Gaikwad, HoD Dr. Rupali Gulalkari and collogues at Chemistry Department, BJS College for their support and encouragement. I hope this booklet will provide an insight of solvothermal and hydrothermal techniques to all the readers.

Thank you,

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Associate Professor in Chemistry, BJS'S ASC College, Pune

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Introduction

“Hydrothermal or solvothermal’ process means accomplishment of chemical reactions in presence of water or any other solvents enclosed in vacuum-packed containers. In this process the temperature of solvents may be taken to nearby solvent’s temperature critical points through heating instantaneously with self-generated pressures.

“Hydrothermal synthesis” is demarcated by the assorted reactions in aqueous medium beyond 100°C, at 1bar pressure. In the hydrothermal technique water is used as solvent while in “solvothermal process” any organic solvents are cast-off[1, 2]

Hydrothermal synthesis is a skill that has gained specific attention in applied sciences in the preceding few years. The current advances in the synthesis of nanomaterials by assorted chemical solution reactions favored the production of a huge mass of innovative functional materials. Furthermore, the particle size control improved by high crystallization kinetics occurring in aqueous substances delivers the adequate circumstances to yield nanoparticles of several inorganic compounds.

The novelty of technology activates the challenges of improving the synthesis of advanced and functional nano-materials to concur the necessities of the up-to-date technology. Hence, the hydrothermal synthesis of nanoparticles (NPs) has arisen as a workable performance to create inorganic nano-materials on a bulky amount in continuous flow reactors at a moderately lower price.

2. A Short Account of the Method

The word, hydrothermal, was used first time in geology during the mid19th era when geo scientist replicated solvothermal states to learn the development of particular crystals and rocks. The solvothermal growth of crystalline quartz was primarily reported in 1945 by Karl Emil von Schafhäut, a German geologist. Thereafter, R. Bunsen described developing crystals of $BaCO_3$ as well as $SrCO_3$ at 200°C under 15 atm pressures by using airtight glass tube in 1848. Thereafter, Henri Hureau de Sénarmont created rock crystal of several minerals via solvothermal synthesis. In 1905, G. Spezia reported the evolution of macroscopic minerals by using various suspensions of sodium silicate in a container lined with silver.

Since the past 30 centuries or so, there has been a firm rise in the numerous of progression strategies and diverse solicitations for NPs made by the use of solvothermal processes. With these solvothermal flow processes, different parameters like temperature, pressure, flow rate, composition etc., can be independently varied and modified to obtain tailor-made nanomaterials. It is observed that scientists have utilized hydrothermal techniques for the synthesis of various products like quartz, zeolites, gemstones, ferrite, carbon allotropes, complex coordinated compounds etc[3,4.]

The hydrothermal techniques provide substantial benefits as they can function at moderately low temperatures with ecofriendly processing routes, without using harmful organic solvents. In a usual hydrothermal route, the solvent H_2O is boiled beyond its steaming temperature by heating under compression for crystallization of resources, analogous to practices in which reserves are made underneath the earth strata above extensive ages deep under the sea. This happens in geologically dynamic areas of the oceanic ground where saltwater breaches clefts in the rock and is then heated by lava.

After some epoch, the water softens and crystallizes out from the rock and as the warm H_2O upsurges; certain of the reserves precipitates out and then strengthen on the perimeter of the duct. Ultimately, a giant chimney-like assembly is molded; named as "black smokers". The temperature of the H_2O approaching out of oceanic ducts can go over 400°C; consequently, there are apparent equivalents to the situations in a lab-based continual reactor. Hydrothermal production is generally used for creating materials in aqueous solution exceeding ambient temperature and pressure.

Hydrothermal synthesis can be described as simulated way to manufacture nanoparticles. It is typically done at an elevated vapor pressure with a high-temperature solution. The solvothermal procedure has existed extremely prevalent one, chiefly in the previous twentieth centuries. The term "hydrothermal" is understandable term, "hydro" stances for aquatic/H_2O and "thermal" means warmth/heat. Sir R. Murchison, a legendary, remained the leading scientist who used this term to clarify the exploit of H_2O at sophisticated temperature and pressure in carrying out variants in the earth's layer foremost to the growth of diverse crystals. Currently, solvothermal technique has created its place in numerous divisions of science and technology, casing a diversity of arenas[5, 6.]

In solvothermal production, manufacture of nanostructures can arise at diverse temperatures: from ambient to higher temperatures. In order to regulate the shape of the ingredients to be arranged, at lower or higher pressure states can be cast-off dependent on the vapor pressure of the core arrangement in the reaction. Some categories of nanostructures have been efficiently produced by the treatment of this tactic. There are several benefits of solvothermal technique over others.

Nanoparticles by means of extraordinary vapor pressures can be created through the solvothermal technique by least damage of resources. The alignments of nanostructures to be manufactured can be well organized in solvothermal production via fluid phase transformations. Luis Brus clarified that solvothermally organized nanoparticles of CdS in suspension, prepared in water had a hypso-chromic shift in the UV-visible absorption spectrum which matched with bulk CdS.

Nanoparticles having size less than the Bohr radius display distinct energy levels comparable to single atoms. Contrasting the band energies detected in bulky resources, each exclusive mineral width on the nano-scale resembles to a distinct energy. Resources that display this distinctive are called "artificial atoms" or "quantum dots" (QDs). Current research reveals that the solvothermal practices are of vital importance in governing the size of the semiconducting materials. Amalgamation of the QDs characteristically necessitates a source of cation which is solvable in the designated solvent and a surfactant that covers or calm down the QDs, fascinating its development. For instance, CdSe QDs are organized by soft solvents as well as coating agents. The blend is heated to 300°C, afterwards selenium in elemental form is dissolved in Tri Butyl Phosphine (TBP)[7, 8.]

The reaction mixture is then quenched and nanocrystals are perceived. ZnO is alternative instance that can be arranged solvothermally and display QD possessions. To achieve this, zinc acetate dihydrate was melted in isopropyl alcohol at about 50 °C. Later, the resultant was refrigerated to 0°C and thereafter sodium hydroxide was added to get the ZnO precipitate. Afterwards, the blend was heated up to 65°C for a period of time before a capping agent (1-dodecanethiol) was inoculated to rest the growth. QDs can be created by the solvothermal process in plentiful forms comprising spheres, sticks, tetrapod and teardrops by governing the heat, concentration and duration. Moreover, a shell of one arrangement (e.g. ZnS) can be generated over a core of new nanocrystals (e.g. CdS). The core can also be cast-off as a seed to produce superior particles by regulating the concentration after the initial growth. Many QDs claims are raised by size and shape control and solvothermal synthesis is the key technology for attaining this control.

Nanomaterials produced by this process are mostly appropriate to biotech solicitations as the nanoparticles are hydrophilic due to superficial hydroxyl groups.

These hydroxyl groups frequently impact the concerned properties of the nanoparticles. Other solvothermal methods, however, can be cast-off to formulate nanoparticles which are made hydrophilic by addition of surfactants. Gold nanoparticles are of specific attention due to their inert nature. Mono-sized gold nanoparticles were set by the solvothermal reduction method analogous to that designated by Chen and Kimura. In this technique, hydrogen tetra chloroaurate tetrahydrate was reduced with sodium borohydride and mercaptosuccinic acid was cast-off as a preservative[9,10.]

3. Devices used in hydrothermal and solvothermal processes

Solvothermal is a technique of creation of solo crystals that is governed by on the solvation ability of reserves in hot H_2O beneath extraordinary pressure. Solvothermal reactions mostly continue in a vacuum-packed device, called as ‘autoclave’, a high-pressure device.

Typically, containers are made-up of metals with Teflon or blend coatings or comprising an additional beaker-like cylinder made from Teflon to guard the autoclave build from the extremely harsh solvents, which is apprehended at great temperature

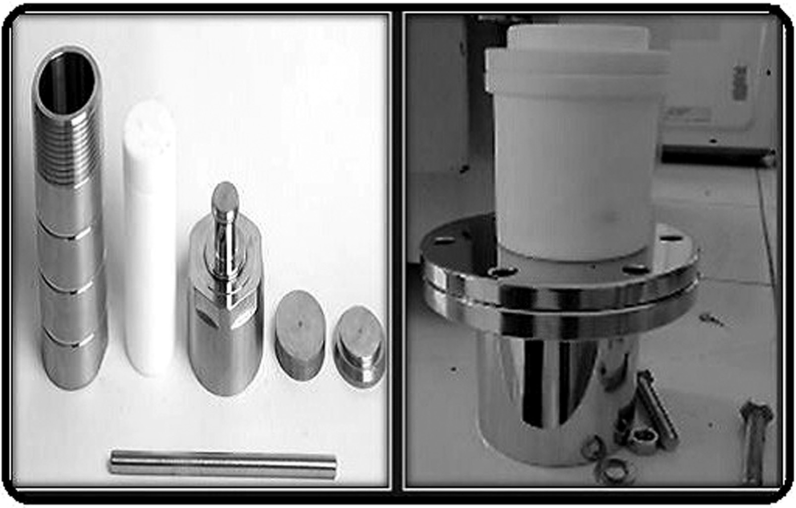

Fig. 1 Hydrothermal reactor

and pressure as shown in Fig1. Under certain circumstances, a Bourdon gauge is fixed to the autoclave to unswervingly screen the pressure and the autoclaves are fitted out with stirring fixtures to lessen the varied concentrations in the reactor. The crystal progression is achieved in device entailing of a steel container under pressure.

The solvothermal process is a capable liquid phase preparation tool that has recognized quickly in modern ages. It is extensively cast-off in several arenas, such as the piezoelectric, ferroelectric, ceramic powder, and oxide film fields. Utmost solvothermal reactions and illustrations are carried out in a sealed reactor. Synthetic quartz crystals manufactured in the autoclave shown in Western Electrics pilot hydrothermal quartz plant in 1959[11, 12.]

Generally, in this process reaction containers are metallic autoclaves with Teflon or alloy coatings to protect from the tremendously corroding solvent that are apprehended at advanced pressure and temperatures.

The autoclaves are usually thickly dense-walled steel cylinders with an airtight lid that can tolerate extraordinary temperatures and pressures for extended timings. Moreover, the reactor substance from which it is made must be non-reactive to the solvent used in process. The closing of Teflon cup is the utmost vital component of the autoclave. Quite a lot of strategies have been established for cap

designs, the greatest well-known being the Bridgman lid. In utmost circumstances, steel-corrosive solutions are mostly cast-off. In order to avoid deterioration due to erosion of the inner void, protective attachments are mostly used in autoclaves. They may have the identical outline as that of reactor fitting in the internal cavity or be “floating” type insertions that conquer only portion of the reactor inside.

Enclosures may be made of metals like Cu, Fe, Ti, Ag, Au, Pt, or materials like quartz or Teflon, reliant upon the temperature. The authentic container is of stainless-steel (SS) recognized as autoclave reactor. It holds Teflon coating inside the SS autoclave or separate Teflon container as shown in Fig.2. Thus it can stand sophisticated temperature as well as pressure above extended time period.

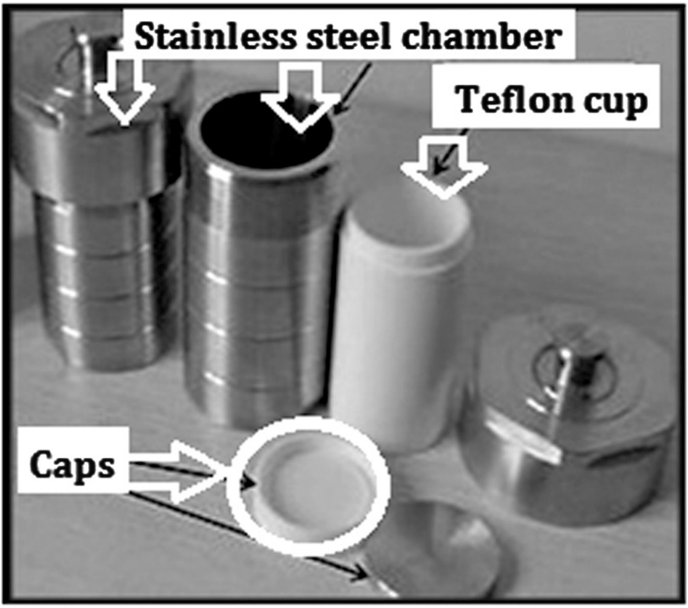

Fig. 2 Hydrothermal Teflon cup with steel chamber

A perfect reactor ought to have the subsequent features:

a. It should be inert to acids, bases, oxidizing agents and various solvents.

b. It must be effortlessly accumulate and dissimulate.

c. It should have adequate size to gain anticipated temperature grade.

d. It must be damp-free at chosen temperature and pressure.

e. It has to stand at elevated pressure and temperature for extended period interval.

4. Fundamental Mechanism:

It is a manifestation progression straight from solutions that characteristically comprises dual stages: crystal nucleation and consequent development. By monitoring process measures like temperatures, pH, concentrations, and additives, the end products may be manufactured with estimated unit dimensions and shapes.

4.1 Procedure: At the beginning, the reacting substances are dissolved and passed in the solution in the form of ions or molecular assemblies. Thereafter, the ions or particles are divided by the temperature variance amongst the higher and lower parts of the reactor. They are then moved to the lower temperature section, where the seed crystal is grown-up to custom a supersaturated solution. Subsequently, the adsorbed substance transfers at the edge. Lastly, the liquefied material grows out in crystalline form. An autoclave must be stress-free to accumulate and take apart, be damp-proof and retain acceptable existence under the investigational temperature and pressure assortment. Commonly various materials are unsolvable in H_2O at ambient circumstances; henceforth it requires raised temperatures and pressures. In solvothermal schemes, the viscidness of water declines and the motion of particles and ions significantly increase, so that crystals propagates speedily under solvothermal circumstances.

Promptness of solvothermal method is primarily organized by several constraints like pH, pressure, temperature and the reaction period. The composites with lower solubility at standard temperature in aqueous solution turn into exceedingly solvable at elevated temperatures in a sealed reaction scheme. Due to advanced temperature, the nucleation turn out to be quicker, resulting consistently spread nano sized units having decent crystal assemblies and morphologies (Fig.3) that results in structures with extremely translucent and exclusive morphologies[13, 15].

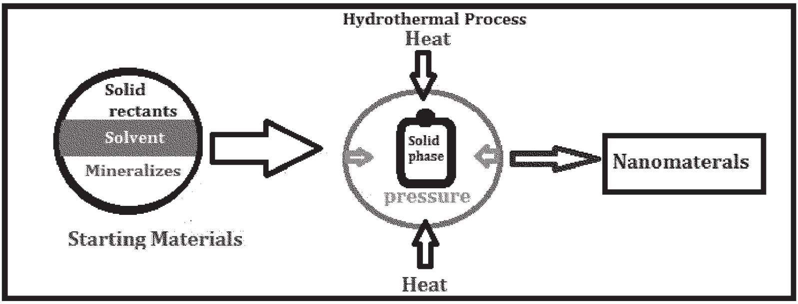

Fig. 3 Hydrothermal process for nanomaterial synthesis

5. Types of hydrothermal techniques

5.1 : Temperature-difference technique

The most widely used technique in hydrothermal synthesis is temperature difference technique. In this method, the super saturation is achieved by reducing the temperature in the crystal growth zone. The autoclave is heated in order to create two temperature zones. The cooler and denser solution in the upper part of the autoclave slopes while the counter flow of solution rises. The solution becomes supersaturated in the upper portion as the result of the reduction in temperature and crystallization sets in.

5.2 : Temperature-reduction technique

In this technique, crystallization takes place without a temperature gradient between the growth and dissolution zones. The super saturation is achieved by a gradual reduction in temperature of the solution in the autoclave. The disadvantage of this technique is the trouble in monitoring the growth process and introducing seed crystals. Hence, this method is very rarely used.

5.3 : Metastable-phase technique

This technique is based on the variance in solubility between the phase to be grown and that serving as the starting material. The solubility of the metastable phase exceeds that of the stable phase and the latter crystallizes due to the dissolution of the metastable phase. This technique is typically combined with one of the other two techniques mentioned above.

6. Synthesis of nanomaterials by hydrothermal technique

Hydrothermal technique is one of the most ordinarily used approaches for preparation of nanomaterials, which involves stages as shown in Fig.4. In this method, the establishment of materials may occur in a widespread temperature choice commencing from ambient temperature to higher temperatures.

In order to regulate the shape and size of the nanomaterials to be produced, either lower or higher pressure settings may be cast-off depending on the vapor pressure of the core material.

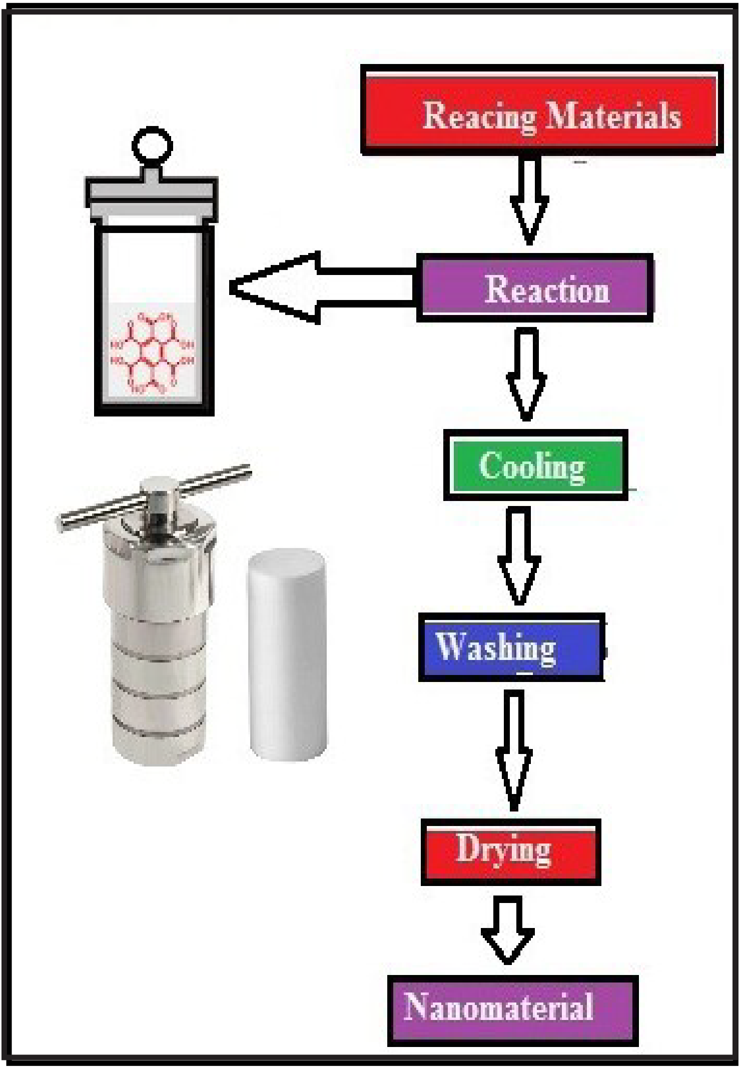

Fig. 4 General steps in hydrothermal synthesis of nanomaterials

Various forms of materials with unique morphologies have been effectively created through the usage of this methodology. There are noteworthy benefits of solvothermal technique over other methods. Nanomaterials with high vapor pressures can be manufactured hydrothermally with slightest harm of resources. The alignments of materials to be created may be finely well-ordered in solvothermal synthesis via liquid or multiphase reactions[16, 17.]

7. Hydrothermal synthesis of Iron oxide nanoparticles

7.1 Method 1: Synthesis of hematite

Aqueous solution of $FeCl_3.6H_2O$ was agitated at around 40 to 50°C for 45min to get a stable solution. The pH is attuned to alkaline (pH=10 to12) by adding adequate quantity of aqueous ammonia and stirred for 60 to 90 min at R.T. to acquire brownish red precipitate. It is then shifted into Teflon lined SS reactor to execute hydrothermal synthesis at optimized circumstances i.e. at about 200 to 250°C for a period of 8 to10 h to accomplish α-Fe_2O_3 nanoparticles. The resultant brick red precipitate is then sieved, washed numerous times with DI water and desiccated in oven at 60 to 70°C for 36 to 48 h to get powder of α-Fe_2O_3 nanoparticles.

$$FeCl_3.\ 6\ H_2O + NH_3\ (25\%) \xrightarrow[\text{6 h, 200°C}]{\text{Hydrothermal}} \alpha\text{-}Fe_2O_3\ (\text{HAM1})$$

Synthesis of hematite nanoparticles

Method 2: An aqueous solution of ferric chloride hexa hydrate was arranged and the solution pH is then attuned to 10 to 12 by addition of suitable quantity of base like trimethyl amine. The blend was stirred for 60 to 90 min, at room temperature and thereafter the mixture is placed in a reactor in adjusted situations at 200 to 250°C for 6 to 8 h. The precipitous obtained was sieved, washed a number of spells with DI water and dried in oven at 60 to70°C for 24 to 48 h to gain dry powder of α-Fe2O3nanoparticles.

$$\mathrm{FeCl_6\ H\ O} \xrightarrow[\text{6 hrs/200 Deg.Celsius}]{\text{Hydrothermal/Trimethyl amine}} \alpha\text{- Fe O}\ \ (\mathrm{HAM_2})$$

Synthesis of hematite

7.2 Hydrothermal-Coprecipitation: synthesis of magnetite (Fe_3O_4)

Method 3: Magnetite (Fe_3O_4) nanomaterial is produced in dualistic stages i.e. coprecipitation followed by hydrothermal method. The ferric chloride hexahydrate, ferrous sulphate heptahydrate are taken in appropriate quantity and melted in DI water.

To this, gentle addition of aqueous ammonia is done with continuous stirring and heating (50 to 60°C) on magnetic agitator till the solution becomes alkaline (pH= 10 to 12). This reaction mixture is then transported into autoclave, for hydrothermal synthesis of magnetite nanoparticles at 200 to 250°C for 6 to 8 h. The blackish precipitate gained is then strained, wash away with DI water and desiccated at 60 to70°C for 24 to 48 h to acquire magnetite (Fe3O4)[18,19].

$$FeCl_3.6\,H_2O + FeSO_4.7H_2O \xrightarrow[\text{NH}_3\text{ (25\%)/6 hrs/200 Deg.Celsius}]{\text{Coprecipitation-Hydrothermal}} Fe_3O4 \text{ nanoparticles}$$

Coprecipitation-hydrothermal synthesis

8. Advantages of the hydrothermal technique

1. A main benefit of this technique is that, it may be mongrelized with additional procedures to achieve rewards such as improvement of reaction speed or the capability to create novel resources.
2. A noticeable effort must be taken to enhance solvothermal synthesis in combination with various other techniques like microwave, ultra-sound, hot-pressing etc.
3. One of the prime benefits of using this technology is usage of H_2O as solvent, for environmental and profitable gains.
4. It is possible to synthesize numerous crystalline materials with higher melting points at lesser temperatures with this technique.
5. By employing this technique fine particles nanomaterials are made straight from the solutions.
6. This one is promising technique for getting controlled particle size and shapes with diverse preparatory ingredients under solvothermal circumstances[20].
7. Use of cheaper precursor substances as compared to expensive materials used in sol–gel method.

8. Starting material that is generally low boiling is likely to condense during the solvothermal course and thus preserve the stoichiometry. Subsequently, extremely wholesome nano powders may be gained.
9. This synthesis technique is completed at low-temperature with several properties attainable straight less than 300°C. The comparatively low temperatures can breakdown stable precursors under pressure, thus avoiding the widespread accumulation of solid-state reactions which typically carried out at higher temperatures.
10. The solvothermal route is beneficial to yield solid solution units with controlled size dispersal, shape and complex chemical composition.
11. The solvothermal manifestation is a self-salvation procedure, throughout which the developing crystals apt to discard contaminations existing in the growing environs. The contaminations are consequently removed from the system together with the crystallizing solution, which does not take place during other production methods.
12. The materials grownup by this method hardly needs calcination stages. This is predominantly important for synthesizing extraordinary quality nanomaterials.
13. Hydrothermal creation is accomplished in a sealed arrangement from which diverse substances can be recuperated and reprocessed, consequently making it an

environmentally gentle technology.

14. This route of synthesis of materials can be effortlessly scaled-up as per industry requests since it offers the chance for profitable and reproducible manufacture of superior powders on huge industrialized scales.
15. These approaches are more ecologically gentle than several other production approaches, which can be ascribed to energy- conserving low processing temperatures, absence of milling, ability to recycle waste and safe and appropriate dumping of left-over that cannot be reprocessed.
16. Materials prepared under hydrothermal circumstances display enhancement in point defects as related to materials set by high-temperature production approaches. The struggle of water assimilation can be avoided by correctly regulating the production conditions otherwise by custom of non-aqueous solvents (solvothermal treating).
17. Additional significant technical benefit of the hydrothermal technique is its competence for incessant materials invention, which can be predominantly valuable in continuous manufacture. These crucial compensations have to be arbitrated contrary to certain disadvantages of the hydrothermal technology.

9. Shortcomings of hydrothermal technique

1. Previous evidences on solubility of starting materials are mandatory.
2. Solvothermal slurries may be destructive.
3. The yields are relatively less than some of the other techniques such as coprecipitation.
4. Accidental explosion of the high pressure container may occur.

10. Recent applications of hydrothermal technique for the synthesis of nanomaterials

The solvothermal and hydrothermal techniques are eco-friendly, low-cost, up-front, with the reactions supported at comparatively low temperatures. These techniques have great control on particle size, in crystallization and morphologies of the materials through reaction time-duration and temperature range, permitting the formation of superior quality nanoparticles.

These techniques are highly effective to manufacture nano-crystals since it exploits the solubility of some inorganic substances at convinced temperatures and extraordinary pressures. The hydrothermal and solvothermal techniques are cast-off to formulate single crystal grains with advanced crystallinity than grains formed in other methods.

Many researchers successfully fabricated various NPs by hydrothermal and solvothermal techniques[23,38].

Few of them are discussed in Table1.

Table1 Hydrothermal and solvothermal conditions used for the synthesis of nanomaterials for focused applications

Sr. No.	**Material**	**Reaction conditions**	**Applications**
01	Functionalized Carbon nano-dots (FCN)	200°C, 24 h, DI water	Bio imaging[24]
02	V_2O_5 nano-fibres	250°C, 0.5 h, DI water	Cathode material[25]
03	Graphene@ V_2O_5	120°C, 24 h, DI water	Enhanced energy storage[26]
04	ZnO	120°C, 24h, DI water, capping agents	VOC gas sensor[27]
05	TiO2 /CuO binary system	180 °C , 12 h. pH=10	Antibacterial properties[28]
06	CdxInyS (x+1.5y) nanocomposites	120°C, 24h, DI water	Photo-catalyst[29]
07	Silicon Hydroxyapatite nano-powders	150°C, 1 h, DI water, MW-hydrothermal synthesis	Various biological applications[30]
08	Sn-beta zeolites	120°C, 24 h	Glucose isomerization[31]
09	Ag-doped ZnO	120°C	MW-assisted hydrothermal Electronics purpose[32]

10	Carbon quantum dots (CQDs)	180°C, 2 h	Sensing applications[33]
11	Ni3TeO6 and Cu3TeO6	180°C , 12 h. pH=7	Magnetic and photo-conductivity applications[34]
12	Fe3O4 nanospheres	200°C, 10 h, ethylene glycol	Glucose sensor[35]
13	Titanium dioxide/ graphene aerogel	200°C, 1h	Photo-degradation of methylene blue[36]
14	Graphene aerogel	80 to 180°C, 4 to 24 h	Oil sorption[37]
15	Magnetite nanoparticles	120°C, 12-24 h	Various applications[38]

11. Future scope of the technique

Solvothermal technique compromises several compensations over traditional and non-conventional man-made procedures. Altogether arrangements of nanomaterials can be created solvothermally. Furthermore, the proficiency to precipitate previously formed precipitates straight from solution controls the speed and consistency of nucleation, development and ageing, which consequences in better governor of dimension and morphology of crystals through considerably reduced aggregation levels. The eradication of masses combined with small unit size dispersions in the starting material leads to optimized and reproducible properties of nanomaterials because of better microstructure control. Solvothermal treatments may be carried out in an extensive diversity of blends of aqueous and solvent mixture-based schemes. As compared to solid-state methods, solutions provide an opening for spurt of dispersal, adsorption, reaction speed and crystallization. On the other hand, unlike various progressive approaches that can formulate enormous diversity of forms, chemicals, the expenditures for instrumentation, energy and starting materials are far less for solvothermal methods. Subsequently, solvothermal creation will discover its viable niche specifically in the capacity of fabricating if technique related and economic difficulties can be effectively astounded in the prospect[21, 23].

Conclusion

- Solvothermal device have been extensively accepted as standard

methods for the construction of nanostructures.

- The method can be exploited to manufacture organic nanoparticles, provided that the carbon-based substances will not be disintegrated at higher temperatures and pressures.
- Hydrothermal equipment stands a superior way in advanced resources treating through its capability to expressively speed up the rate of synthesis.
- Scale-up of the incessant hydrothermal devices is being commenced to yield significant extents of NPs that will

permit industrialized and educational consumers to assess the compensations of the technology.

- The incessant practice is preferably of custom for extraordinary quantity nanoparticles production and innovation. Synthetic zeolites are amongst the utmost frequently man-made nanomaterials by means of hydrothermal processing. Their development strongly depends upon the presence of surface activation agents in the solution. These agents govern the morphology and shape of numerous metal oxide products.

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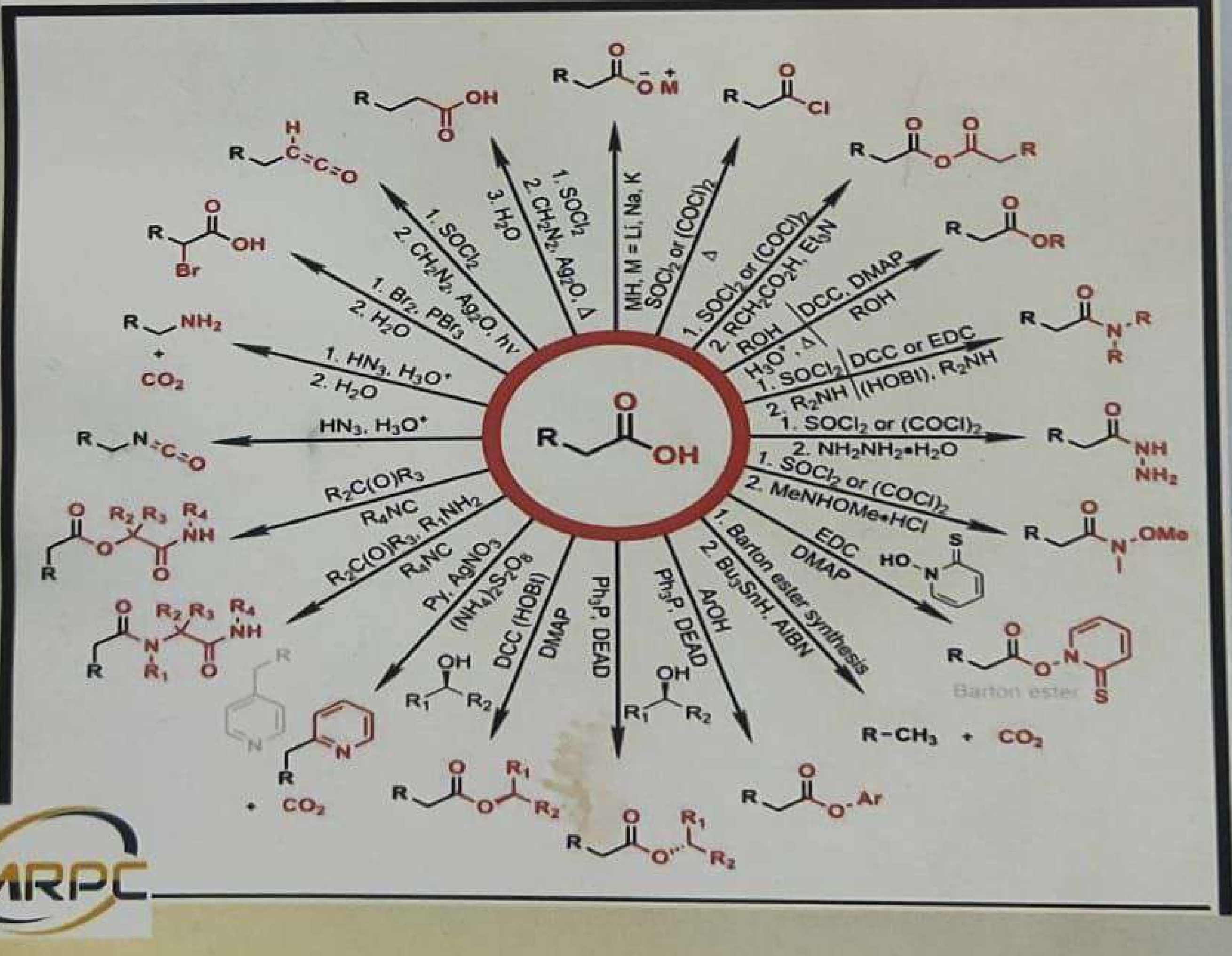

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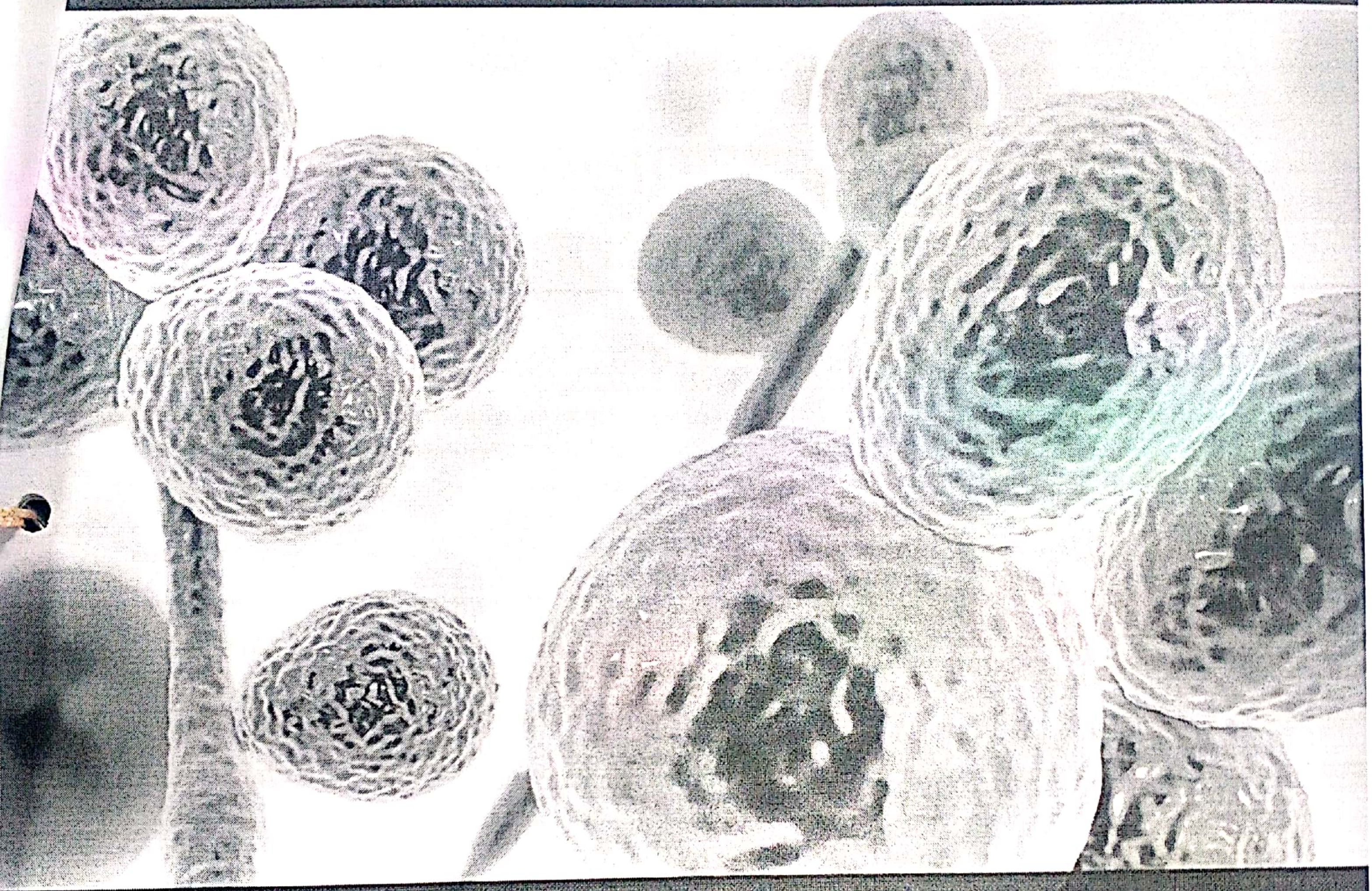

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Rehabilitating Agricultural Soils

Edited by
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Microbiome Research in Plants
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17

Potential use of microalgal metallothioneins and phytochelatins in bioremediation

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1. Bioremediation

The advancement in technology and improved living standard of human beings creates a need of large quantity of new materials as well as energies. These are responsible for the production of waste, results into environmental degradation and destruction of natural environmental components (Mani & Kumar, 2014; Saha et al., 2021). Rapid growth in industrialization, urbanization, mining, and use of pesticides and fertilizers for a prolong time are the primary sources of harmful and toxic pollutants such as heavy metals (HMs), pesticides, microplastics, hydrocarbons, etc. (Emenike et al., 2018; Guo et al., 2020; Rajendran et al., 2021; Xu et al., 2018). Bioaccumulation of these contaminants in food chain causes serious damage to the ecosystem (Zerizghi et al., 2020) and having harmful effects on human body, may lead to cancer, kidney diseases, atherosclerosis, hypertension, Alzheimer's diseases, cardiovascular diseases, etc. (Ahern et al., 2011; Bernhoft, 2012; Flora et al., 2012; Lee et al., 2017; Muszynska et al., 2015; Nawrot et al., 2006). The utilization of microbial source acting as an effective tool for remediation of environment contaminated with toxic organic pollutants and heavy metals (Liu et al., 2020). The bioremediation technique involves the use of microorganisms like microalgae and bacteria to detoxify, transform, or remove HMs and toxic pollutants from environment (Cepoi et al., 2022; Hadiani et al., 2018; Khan et al., 2019; Lopes et al., 2021). Nowadays, more preference is given to these biological-based strategies over other physicochemical approaches, because its implementation is easy and simple, applicable to large areas, more economic, reliable, and ecofriendly (Ashraf et al., 2019). According to the study, cleaning of metal-polluted soils and chemical treatment costs around 100–500 USD per ton, whereas for bioremediation, it costs around 15–200 USD per ton (Meier et al., 2012). Basically, bioremediation process is divided into "*in-situ*" and "*ex-situ*" strategies (Lombi & Hamon, 2005). In "*in-situ*" process, bioremediation takes place at the contamination site, whereas in case of "*ex-situ*," the excavation of these contaminants is performed from its original site and treated elsewhere (Das & Mukherjee, 2007). The "*in-situ*" bioremediation process includes bioventing, bio-

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sparging, bioaugmentation as well as biostimulation (Atlas & Phillip, 2005; Crivelaro et al., 2010; Sharma et al., 2012; Thapa et al., 2012). The "*ex-situ*" process includes the techniques like land farming, composting, biopiles, and use of bioreactors (Chatterjee et al., 2008; Chikere et al., 2011; Soccol et al., 2003; Wu & Crapper, 2009). Both the processes are based on the principles biodegradation, immobilization, biotransformation, removal, or decontamination of various environmental pollutants by using microorganism and plant sources (Abatenh et al., 2017). The chemical contaminants are used by microbes as an energy source and are metabolized into useable energy via redox reactions. The resulting metabolites and by-products are less harmful than original pollutants which are releasing back to the environment (Nester et al., 2001).

2. Heavy metals and their hazardous effects

Heavy metals are considered as most threatening environmental contaminants (Gustin et al., 2021). HMs are the elements with atomic number and density greater than 20 and 5 g/cm^3, respectively (Ali & Khan, 2018). These are categorized into three classes like toxic (a) HMs (e.g., Pb, Hg, Zn, Cu, As), (b) precious metals (e.g., Ag, Pt, Pd, Au), and (c) radionuclide HMs (Th, U, Ra, and Am) (Wang & Chen, 2009). HMs are also classified based on biological perspectives into essential as well as harmful elements. Essential metals or metalloids are micronutrients obtained from plant and animals such as Cu, Zn, Ni as well as Fe, but they can prove toxic above certain thresholds (Garcia-Garcia et al., 2016). On the other hand, toxic elements are nonessential metals, considered to be toxic even at low concentrations (Asad et al., 2019; Ashraf et al., 2019). Nonessential HMs are having serious toxicities toward microorganisms, plants, animals as well as human beings even at very less concentrations (Ali et al., 2019). HMs are not biodegradable, and its toxicity and accumulation in soil and environment attracted much attention worldwide (Kandziora-Ciupa et al., 2021). These can cause direct and indirect effect on the growth of plant like injury to the root, decrease in concentration of carotenoid, necrosis, chlorosis, inhibition of enzymatic activities, decrease in activities related to photosynthesis, and nutrient imbalance (Hasan et al., 2017; Lewis et al., 2001; Mascher et al., 2002; Sachan & Lal, 2017; Shaibur et al., 2009; Yadav, 2010). The bioaccumulation of HMs results into pass the food chains to human beings causing serious and adverse effects on health. HMs can enter the human body by ingestion, through food chain, drinking contaminated water, and contact with environmental contaminants. Its exposure for several years leads to the dangerous effect on the health of human beings (Kumar et al., 2019; Njoga et al., 2021). However, exposure to HM continues and arising in some regions (Järup, 2003). The industrial and domestic waste disposal containing HM threatens the aquatic organism and damage to aquatic ecosystem (De Filippis & Pallaghy, 1994). Hence, the treatment of water contaminated with HMs becomes a global issue and research area of interest for scientists and environmentalists. It becomes necessary to remove these contaminants from aquatic ecosystem as well as industrial wastewater in order to protect environmental as well as human health (Denouche et al., 2021). The use of traditional technologies (e.g., lime precipitation and ion exchange) to remove the HM is often expensive and ineffective (Wilde & Benemann, 1993). So, it becomes essential for searching the newer technologies for the remediation of HMs, which should be economic, effective, and consistent, shall reduce the heavy metal concentration up to significant, and promising approach to remove the HM contaminants (Fu & Wang, 2011; Sheng et al., 2004).

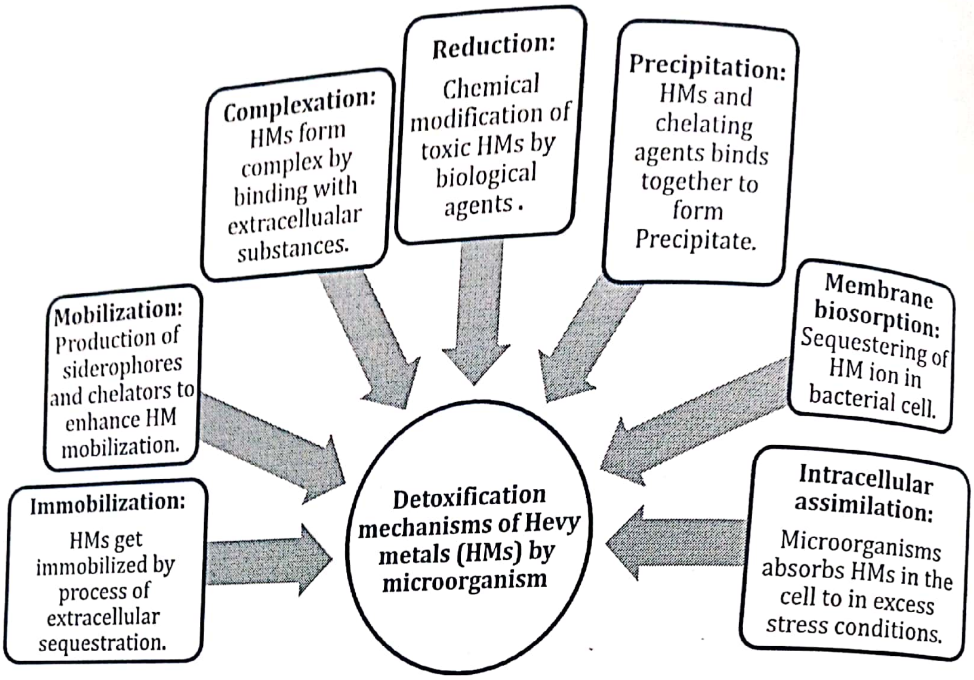

FIGURE 17.1

The mechanisms for detoxifications of heavy metals (HMs) from the soil using microorganisms.

3. Heavy metals detoxification mechanism

The HM accumulation in the cell leads to cell homeostasis disorders, causes damage to chloroplast and pigments, affects the structure and function of DNA, and finally the cell destruction takes place by the formation of reactive oxygen species (ROS) (Kumar et al., 2016). Microorganisms are playing important role in detoxification process which can precipitate, sequester, biosorb with change in oxidation states of different metals (Rizvi et al., 2020). The fundamental mechanism of detoxification of HM involves their chelation in the cytosol by certain ligands like metallothioneins (MTs), phytochelatins (PCs), organic acids, and amino acids which reduce the intracellular contents of HMs and their phytotoxicity (Haydon & Cobbett, 2007). The microbial conversion of toxic metals into less toxic takes place by using the microbial enzymes such as oxidoreductases, dioxygenases, and peroxidases (Saha et al., 2021). The mechanisms involving utilization of microorganism for detoxification of the HM from polluted soil is represented in Fig. 17.1.

4. Metallothioneins

MTs are metal-binding proteins characterized by Vallee et al. in the year of 1950. It is a product of mRNA translation, characterized as cysteine-rich protein with molecular weight (6–7 kDa). The induction of MTs in aquatic organism plays a role of significant biomarker for heavy metal toxicity as well as bioaccumulation (Stillmann, 1995; Won et al., 2008). MTs play a significant role in essential metal supply to the cells and carrying out the transport of harmful metals to other cell organelles (Capdevila & Atrian, 2011). Basically, MTs are small proteins (≤300 amino acids) consisting of some aromatic residues (<10%) and high amount (15%–35%) of cysteine, which coordinate metal ions

(Ziller & Fraissinet-Tachet, 2018). MTs are classified into four types, having tertiary type of structure with metal-binding property is explained for type 4 plant MTs. Structurally type 4 plant MTs consist of cysteine-rich shorter N-terminal acting as domain for metal-binding and long C-terminal domain containing MTs (Leszczyszyn et al., 2013) along with shape having HM-MT complex varies as per total numbers of cations present in bound form (Bell & Vallee, 2009). MTs consist of 15 distinct families, and each family comprises evolutionary respective proteins present in organisms of similar taxonomic groups. The nine conserved motifs which are cysteine rich are present across various MT families like CCXCC, CXCXCXC, CCC, CCXC, CXCC, CXCXC, XCCX, CXC, and XXCXX. The eukaryotic MTs are identified majorly in plants, ciliates, fungi, and metazoan (Ziller & Fraissinet-Tachet, 2018). Most of the MTs obtained from eukaryotes of microorganisms are associated with ciliates (e.g., Paramecium, Tetrahymena), Apusozoa (Thecamonas), and parasitic Apicomplexa (e.g., Theileria, Babesia) or Amebae (Entameba), as well as six types of microalgal genera (Thalassiosira, Symbiodinium, Chlorella, and Aureococcus) include marine type of representatives (Balzano et al., 2020). MTs are the molecules that chelate the toxic metals like Cd, and thus reduce the cytotoxic, free metal ions concentration. Some of the MTs are involving in Zn as well as Cu homeostasis. The heavy metals like Pb, Cu, Zn, As, Cd, Au, and Ag are responsible for inducing the class III MT biosynthesis within several microalgae like *Stichococcus bacillaris*, *Stichococcus tenue*, and *Stichococcus subspicatus* (Perales-Vela et al., 2006; Robinson, 1989).

5. Phytochelatins

PCs are cysteine-rich peptides with metal-binding properties consisting three amino acids: glutamate, cysteine, and glycine, as well as sulfhydryl group of cysteine involves in metal sequestration (Danouche et al., 2021). PCs are also referred as class III MTs identified primarily in higher plants (Cobbett & Goldsbrough, 2002). PCs are synthesized from γ-glutamylcysteine and the synthesis is catalyzed by transpeptidase enzyme, PCs synthase, which needs posttranslational activation by heavy metals (Chen et al., 1997; Grill et al., 1989; Hayashi et al., 1991; Torres et al., 2008). The biosynthesis of glutathione (GSH) includes two reactions: γ-glutamylcysteine formation from cysteine and glutamic acid, the reaction catalyzes by enzyme glutamate-cysteine ligase (GCL) and glutamylcysteine ligation with glycine to form GSH, the reaction is catalyzed by GSH synthetase (GSHS) (Musgrave et al., 2013). In the final step, GSH binds to other glutamylcysteine units to form PCs. PCs are able to form a complex or chelates through thiol group present in cysteine residues side chain. HM cations can coordinate up to four sulfide groups from one or more PCs within HM-PC chelates (Hirata et al., 2005). Most of the metals and metalloids like Cu, Zn, As, Pb, and Cd can be helpful for the PC synthase activation both in vivo as well as in vitro. Earlier PC production is reported within freshwater algae as a response to heavy metals present in the mining water (Pawlik-Skowronska, 2001).

6. Use of microalgal metallothioneins and phytochelatins in bioremediation

MTs are known as a superrich cysteine family present in cytosol, which is characterized by low molecular weight gene-encoded proteins. These are metal-binding proteins having significant role for controlling the intracellular concentration of metals with regular level. The majorly known MTs from microalgae belongs to Chlorella Symbiodinium, Aureococcus, Thalassiosira, Nannochloropsis, Ostreococcus genera (Balzano et al., 2020; Gaur & Rai, 2001). The microalgae that synthesize enzymatically PCs rather than MTs in response to metal exposure. The synthesis of PCs in microalgal strains is primarily identified in *C. fusca* on exposure to Cd(II) ions (Gekelar et al., 1988). From research, Hg−PCs are identified in *C. sorokiniana* with mercury exposure, and synthesis of PCs also found in Cu(II)-treated *Scenedesmus bijugatus* and Pb(II)-treated *S. bacillaris* (Gomez-Jacinto et al., 2015; Nagalakshmi & Prasad, 2001; Pawlik-skowron, 2002). Cd(II) is found to be a significant stimulator of PC synthase in Chlamydomonas species. PC synthesis is majorly induced by Zn present in Dunaliella species. The electron-dense material is found in vacuoles of green alga *Tetraselmis suecica* which is exposed to Cd(II) as well as diatom *Skeletonema costatum* that accumulated Cu(II) and Cd(II) (Abboud & Wilkinson, 2013; Nassiri et al., 1997; Perales-Vela et al., 2006; Wang et al., 2017). In other study, three microalgae isolated from *P. ambiguum*, *P. typicum*, and *S. quadricauda*, evaluated for the removal and tolerance of mercury, lead, and cadmium present in aqueous solutions. It is reported that cytoplasmic metal concentration is minimized by the formation of complex of metal ion with PCs in the form of metallo-iron, metallo-sulfur, or metallo-phosphate chelates in the cytosol. These are carried into the vacuoles, where acidic pH displaces the metals and allows peptides to return to the cytosol. This mechanism is referred as cellular protection or detoxification mechanism (Shanab et al., 2012).

7. Factors responsible for affecting bioremediation

The prime factor responsible for affecting bioremediation is site characteristic, and other factors viz. pollutant bioavailability, amount of moisture, nutrient availability, pH, temperature, and amount of water also affect the bioremediation efficiency (Leong & Chang, 2020). The type of contaminants and the extent at which it is present in the location or site affect bioremediation. It can be managed by sufficient prior investigation (Abatenh et al., 2017). The high temperature is responsible for the destruction of bacterial cell metabolic activities and can affect the bioaccumulation process. However, pH 6.5−8.5 is considered maximum potential bioremediation (Abatenh et al., 2017; Javanbakht et al., 2014). Within cold environment, nutrient supply in required quantity increases the microbial metabolic activities that results into rise in the rate of bioremediation. Moisture is also responsible for affecting pollutant metabolism rate by influencing osmotic pressure of aquatic sites (Couto et al., 2014). In case of plant-based bioremediation, the factors for the selection of suitable plant include the root system, above-ground biomass, and plant growth. For the growth and metabolism of microorganisms, the water activity values should be 0.9−1.0, and most number of bacteria can grow with upper limit values of water activity (Azubuike et al., 2016; Sharma, 2019). The bioavailability of contaminant is controlled by different process like diffusion, desorption, sorption, dissolution, etc. It can be managed by the application of complexing agents like ethylene diamine tetraacetic acid (EDTA), citric, acetic, and malic acid that form chelates with HMs with increase in bioavailability (Sarwar et al., 2017).

8. Strategic approaches in remediation

The first strategic approach in bioremediation is the use of immobilized microalgae and metals. It is one of the significant approaches in detoxification and metal recovery process. It is reported that the total removal of Cu(II) by calcium alginate gel immobilized *C. vulgaris* is higher than that of agarose *C. vulgaris* system (Aksu, 1998; Hameed & Ebrahim, 2007). In order to increase the specificity toward HM and microalgal metal-binding capacity, the transgenic approach is developed for the use of microalgae for the treatment of heavy metals in wastewater as well as sediments. It is characterized by enzymatic overexpression of which metabolic residues or products enhance the effect of heavy metal-associated stress, heavy metal-binding proteins on the surface as well as in the transgenic cells cytoplasm (Rajamani et al., 2007). Another approach in remediation is metal desorption; algae are used as characteristic biological adsorbents in different restoration processes. The metal that is sorbed on microalgal biomass is desorbed by using solution used for desorption and by which allowing the reuse of biomass in multiple cycles of sorption—desorption (Lu et al., 2006). Recycling of microalgal biomass is also a strategic approach in remediation in which flocculating agent chitosan is used for microalgal recovery. The gel entrapment by the use of synthetic polymers like polyacrylamides, polyurethanes, or natural polysaccharides like agar, alginate is also recommended (Monteiro et al., 2012).

9. Metallothioneins and phytochelatins in heavy metal phycoremediation

Phycoremediation is a type of bioremediation, related to the utilization of algae for removing or mitigation of toxic pollutants (John, 2003). In this process, the HMs are removed from polluted water as well as sediments by using microalgal ability to get incorporated into the metal cations from surroundings. HM uptake process is driven with the use of electrochemical affinity which starts with the adsorption of metal. Electrochemical affinity occurs within metal cations and groups of cell wall polymers that are polar in nature. The dead microalgal biomass can also be used which prevents the risk of contamination of ecosystem, particularly suitable for polluted environments in which the inhibition of microalgal growth takes place (Kumar et al., 2015; Arıca et al., 2005). The accumulation and intracellular transport of HMs in the particular organelles are regulated with use of MTs, PCs, and different heavy metal-binding molecules like polyphosphates. The biosorption rate of HM can be enhanced by adjusting the physicochemical conditions like pH, temperature to which microalgal substrates as well as HMs exposure takes place; as increase in the bioaccumulation of HMs, the scientist expressed recombinantly import-storage systems that consist of primary active transporters and secondary carriers and channels (Diep et al., 2018).

10. Current developments in bioremediation and other applications

Bioremediation is one of the promising and innovative techniques of waste management which use living organism for the removal or neutralization of contaminants (Mosa et al., 2016). Phytoremediation is the efficient, cost-effective, solar-powered, and green alternative technology which is also called as green remediation, botanoremediation, or agroremediation related to use the plant to transform, reduce, extract or immobilization of contaminants present within soil, sediment as well as groundwater (McGrath et al., 2001; Ullah et al., 2015). Another technology of bioremediation includes phytoextraction, phytostabilization, phytovolatilization, phytotransformation, and phytofiltration. Phytoextraction is also called as phytoaccumulation which is related to the use of hyperaccumulating plants which are responsible for the uptake of metal from soil with the use of its roots and accum them in its aerial part (Oladoye et al., 2022). Phytostabilization is also an emerging technology refers to use plant to immobilize soil contaminants through adsorption on or precipitation in root zo (DalCorso et al., 2019). Phytovolatilization is related to the uptake of toxic soil contaminants by pla and its transformation into the volatile products, followed by their discharge in environment (Ali et a 2013). Phytotransformation or phytodegradation is restricted to eliminate the organic contaminants heavy metals are nonbiodegradable (Singh et al., 2018). Phytofiltration technique consists the proce of elimination of organic as well as inorganic pollutants from the water with aid of plant roots (rhi zofiltration), plant shoots (caulofiltration), or seedlings (blastofiltration) (Hakeem et al., 2020). The nanoparticles are having special characteristic that includes improved process of catalysis and adsorption with increased reactivity. Recently the microorganisms and its extracts are proved to be promising, ecofriendly catalysts for engineered nanomaterial. The combination of nanotechnology and bioremediation is called as nano-bioremediation which is more ecofriendly, safe, more economic, and green (Hidangmayum et al., 2022).

11. Future technologies in bioremediation

Biotechnological interventions which primarily include the genetic engineering, for example, various steps which are rate limiting in known pathways of metabolism can be altered and genetically manipulated in order to increase the rate of biodegradation or by the introduction of completely newer pathways of metabolism in microorganism for high amount of HMs accumulation. The study related to hologenomics of microorganisms from plant source which assists for the manipulation of microbial niches helps to increase in resistance against the harmful contaminants. Though there are various technologies available for bioremediation, there is need to develop more suitable ecofriendly technology for the treatment of the multistressed and multimetal-contaminated soil (Saha et al., 2021). Metagenomic perspectives shall also be focused on microbial evolution during the process of bioremediation (Raklami et al., 2022).

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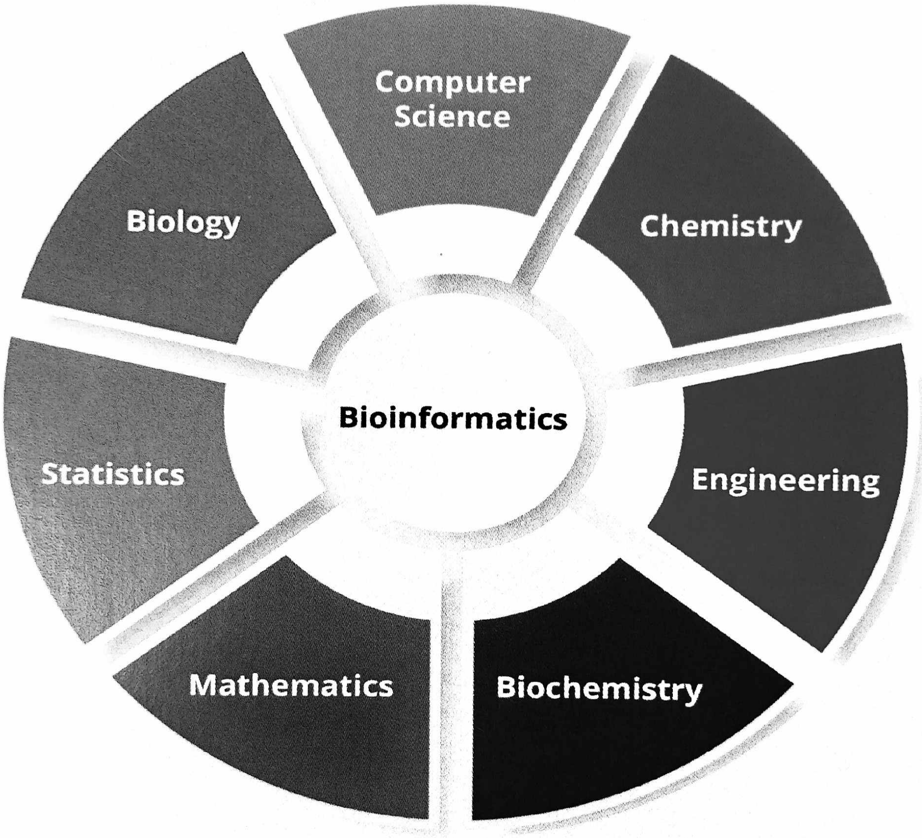

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