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Review Article

Microwave-Assisted Solubility Enhancement Techniques: A Review

Patwekar Shailesh, More Geeta*, Navkhande Yuvraj, Walale Vaibhav

Department of Pharmaceutics, School of Pharmacy, Swami Ramanand Teerth Marathwada University, Vishnupuri-431606 Nanded, Maharashtra, India.

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ABSTRACT

Solubility enhancement of poorly water-soluble drugs remains a critical challenge in pharmaceutical development. Microwave-assisted techniques have emerged as a promising approach to address this issue due to their rapid, energy-efficient, and uniform heating mechanisms. These methods improve drug solubility and dissolution rates through various formulations, including solid dispersions, nanosuspensions, and inclusion complexes. The use of microwave irradiation facilitates molecular dispersion and amorphization of drug particles, enhancing their bioavailability. This review comprehensively explores the principles, mechanisms, advantages, and limitations of microwave-assisted solubility enhancement. Furthermore, comparisons with conventional techniques and potential applications in drug delivery systems are discussed, highlighting the method's growing relevance in modern pharmaceutical research.

INTRODUCTION

Solubility is defined as the ability of a solute to dissolve in a solvent to form a homogeneous system. Temperature, pressure, and the solvent employed all have a basic impact on a substance's solubility. The saturation concentration, at which adding more solute does not raise its concentration in the solution, is a measure of a substance's degree of solubility in a particular solvent.(1) Poor water solubility remains a major hurdle in oral drug formulation, especially for novel chemical entities with high permeability but limited absorption beyond the upper small intestine. This narrow absorption window leads to reduced bioavailability if the drug isn't released promptly in the GI tract. Enhancing solubility and drug release is thus crucial to improve absorption and minimize side effects. Solid dispersions offer a promising strategy by dispersing poorly soluble drugs in hydrophilic carriers, significantly improving wettability, dissolution rate, and local

***Corresponding Author:** More Geeta
Address: *Department of Pharmaceutics, School of Pharmacy, Swami Ramanand Teerth Marathwada University, Vishnupuri-431606 Nanded, Maharashtra, India*
Email : geetamore095@gmail.com

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saturation solubility. These systems, often reducing drug particles to near-molecular levels, are key to boosting oral bioavailability.(2)

Microwave-Assisted Solubility Enhancement is an advanced technique that uses electromagnetic waves (0.3–300 GHz) to generate heat through molecular oscillations within materials.(3) Unlike conventional heating, which warms surfaces first, microwave heating uniformly heats the entire volume by directly interacting with dipolar molecules.(4) Efficiency depends on a material's ability to absorb microwave energy, especially near the molecules' resonance frequency. In organic chemistry, microwave irradiation offers advantages such as rapid volumetric heating, minimal surface scorching, targeted heating, energy efficiency, and low operational cost.(2)

According to the Noyes-Whitney equation, reducing particle size increases surface area and enhances the dissolution rate. However, below ~1 µm, particle curvature also raises dissolution pressure and solubility, as explained by the Ostwald–Freundlich and Kelvin equations. Nanonization, targeting particles between 100–1000 nm, improves the solubility and bioavailability of BCS Class II drugs by increasing both dissolution rate and saturation solubility. Techniques like high-pressure homogenization, jet milling, and nanoprecipitation are used for nanosizing. As particle size decreases, surface energy and Gibbs free energy increase, requiring external energy and stabilizers (ionic, steric, or polymeric) for stability. (5)Advanced solubilization approaches now focus on forming molecular or nanoscale drug dispersions in stabilizing media, with microwave irradiation emerging as a sustainable and effective method for creating such solid-state systems.(6)

The Direct Fusion method melts drug and polymer at high temperatures, risking drug degradation, while the Solvent method avoids heat but involves costly solvents, incomplete solvent removal, and potential stability issues. Microwave-assisted fusion offers a superior alternative by using lower, rapid heating, minimizing drug degradation, shortening preparation time, and eliminating the need for solvents, making it ideal for enhancing drug solubility.(7)

1.1 Need of Solubility

There are several reasons why medication absorption from the GI tract may be restricted, but the two main ones are the drug's poor water solubility and membrane permeability. Before an active substance to pass through the GIT's membranes and enter the systemic circulation, it must first dissolve in the stomach and/or intestinal fluids. Therefore, increasing the solubility and rate of dissolution of medications that are poorly soluble in water are two areas of pharmaceutical research that concentrate on increasing the oral bioavailability of active substances. A scientific framework known as the BCS is used to categorize medicinal substances according to their intestinal permeability and water solubility. Since drug release from the dosage form and solubility in stomach fluid—rather than absorption—are the rate-limiting steps for BCS class II and IV medications, improving solubility will raise the bioavailability of these medications. Table 1 discusses the classification system with examples of various drugs in the following (Table No.1).(8)

Table 1: Biopharmaceutical Classification of Drug

BCS Class	Solubility	Permeability	Examples
Class I	High	High	Metoprolol, Propranolol

Class II	Low	High	Ketoconazole, Griseofulvin
Class III	High	Low	Cimetidine, Ranitidine
Class IV	Low	Low	Hydrochlorothiazide, Furosemide

1.2 Principle of Microwave Assisted Synthesis Process

A] Heating mechanism:

The heating of the mix of materials occurs owing to the interaction of electric field formed by wave and the charge particles when irradiated with high-frequency electromagnetic waves. (9)

B] Dipolar polarization:

Dipolar polarization refers to the heating of material system by polar molecules. The polar molecules likely to orient themselves with an electromagnetic field at the proper frequency. The process results in random motion of molecules due to which the interaction or collision between molecules takes place and produces heat. Polar molecules are resonated and oscillated by microwaves with frequencies between 0.3 and 30 GHz, which improves intermolecular interactions.

C] Interfacial polarization:

The interfacial mechanism is mostly used in material systems formed by conducting insertion of one material into another, which are composed of inhomogeneous dielectric materials. (11)

D] Conduction mechanism:

This process uses resistance brought on by an interruption in the electric current's flow to generate heat within the material system. The oscillation of electrons and ions in the conducting material caused by electromagnetic waves is the primary cause of electric current generation. The heating action causes internal resistance to the generated current.(10)

1.3 Mechanism of Solubility Enhancement

Methods for Improving Solubility

Early in the drug discovery process, formulation methods are necessary when a substance's solubility in aqueous media is restricted. These strategies are still crucial for the selection of lead chemicals and the creation of commercial pharmacological products.

To increase the solubility and dissolution rates of medications that are poorly soluble in water, a number of methods have been employed, including the following:

1) Reduction of particle size
2) Nanonization
3) Cosolvency
4) Hydrotropy
5) pH adjustment
6) Sono crystallization
7) Supercritical fluid (SCF) process
8) Solid dispersion
9) Inclusion complexation
10) Self-Micro Emulsify
11) Liquisolid Techniques
12) Microwave Irradiation Method
13) Microwave Induced Solid Dispersion Method

1) Particle Size Reduction

Drug solubility and particle size are frequently inextricably linked; the ratio of surface area to volume rises with decreasing particle size. Greater contact with the solvent is made possible by the bigger surface area, increasing solubility.(12) The active component is broken down by mechanical stress in traditional particle size reduction

techniques like comminution and spray drying. (13)Thus, a cost-effective, repeatable, and efficient method of improving solubility is made possible by particle size reduction. However, the mechanical forces involved in comminution, like grinding and milling, frequently cause the drug product to experience high levels of physical stress, which could lead to degradation.(14)

Another common method for reducing particle size is micronization. Through an increase in surface area, micronization speeds up the pace at which pharmaceuticals dissolve; it has no effect on equilibrium solubility.(15) These medications dissolve more quickly when their particle size is reduced since this increases the surface area. Drugs are micronized utilizing milling processes such as jet mills, rotor stator colloid mills, and so on. Since micronization does not alter the drug's saturation solubility, it is not appropriate for medications with a high dosage number.(16)

These procedures were used for fenofibrate, progesterone, griseofulvin, and spironolactone diosmin. Micronization enhanced the digestive absorption of each medication, which in turn enhanced its bioavailability and therapeutic effectiveness. The solubility of micronized fenofibrate in 30 minutes of biorelevant media increased by more than ten times (1.3% to 20%).(8)

2) Nanonization

Many Nanonization techniques have recently been developed to improve the bioavailability and dissolving rates of many medications that have low water solubility. The study and application of materials and structures at the nanoscale level, or less than 100 nm, is generally referred to as Nanonization. Drug solubility and pharmacokinetics can be enhanced by Nanonization, which may also lessen systemic adverse effects. Oral bioavailability increase through micronization is insufficient for many novel chemical entities with very poor solubility since the micronized product has a tendency to agglomerate, reducing the effective surface area for dissolving. Nanonization is the next stage. Drugs can be nanonized using a variety of methods, such as spray drying, emulsification-solvent evaporation, wet milling, homogenization, and pear milling. There are numerous instances of medications being nanonized.(8)

3) Co-solvency

Cosolvency is the phenomena where a solute is often more soluble in a mixture of solvents than in a single solvent. Cosolvents are solvents that improve a drug's water solubility. Ethanol, propylene glycol, glycerin, and polyethylene glycols (PEG 300 and PEG 400) are examples of water-miscible cosolvents that are frequently utilized. When creating liquid dose forms like syrups, elixirs, injections, creams, and lotions, this idea is commonly used. Furthermore, additional solvents are used, including benzyl alcohol, dimethyl sulfoxide (DMSO), dimethyl acetamide (DMA), and dimethyl formamide (DMF).(17)

4) Hydrotropy

A solubilization process known as Hydrotropy occurs when a significant amount of a second solute is added, increasing the solute's water solubility. Numerous poorly water-soluble medications have been shown to have their aqueous solubilities improved by concentrated aqueous hydrotropic solutions of sodium benzoate, sodium salicylate, urea, nicotinamide, sodium citrate, and sodium acetate.(8)

Increasing medication solubility without chemically altering the molecule is possible through cosolvency and Hydrotropy, especially for drugs with low water solubility. While

Hydrotropy improves solubility by adding high amounts of particular agents that interact with the drug without changing the solvent, cosolvency involves changing the entire solvent environment using modest amounts of water-miscible solvents. Cosolvency is economical for small-scale formulations, but because of its possible toxicity, it has a higher chance of drug precipitate upon dilution or solvent evaporation, and it might be environmentally problematic. While Hydrotropy is typically more accessible and less costly, it provides superior stability with non-volatile chemicals, is generally safer, and scales better for applications.(17)

5) pH Adjustment

The straightforward and widely used technique to improve the water solubility of ionizable compounds is to alter the ionization behaviour by adjusting the pH of the microenvironment. According to the Handerson-Hessel batch equation and the pH-partition theory, a compound's ability to ionize depends on the drug's pKa and the media's pH. A salt may develop in situ as a result of the alteration in the ionic environment. Therefore, it is impossible for unionized chemicals to create salt. In the gastrointestinal tract, salt production may correspond to the corresponding acid or base forms.(18)

6) Sono Crystallization

To decrease particle size, liquid solvents and antisolvents have also been effectively used to recrystallize weakly soluble materials. Sono crystallization is a new method for reducing particle size based on crystallization utilizing ultrasonography. In order to induce crystallization, Sono crystallization uses ultrasonic power with a frequency range of 20–100 kHz. In addition to increasing the rate of nucleation, it is a useful tool for reducing and managing the size distribution of the active medicinal components. Ultrasound in the 20 kHz–5 MHz range is used in the majority of applications.(8)

7) Supercritical fluid (SCF) Process

Additionally, the number of technologies and applications incorporating supercritical fluids has increased rapidly. The ability of supercritical fluids (SCFs) to dissolve non-volatile solvents has been understood for over a century. The most common SCF is carbon dioxide, which has a critical point. It is affordable, safe, and good for the environment. SCFs are appealing for pharmaceutical research because of their low operating conditions (temperature and pressure). Above its critical temperature (Tc) and pressure (Pc), a SCF exists as a single phase. Due to their intermediate characteristics between pure liquid and gas (i.e., liquid-like density, gas-like compressibility and viscosity, and higher diffusivity than liquids), SCFs have qualities that are helpful for product processing.

Carbon dioxide, nitrous oxide, ethylene, propylene, propane, n-pentane, ethanol, ammonia, and water are examples of supercritical solvents that are frequently employed. The drug particles may recrystallize at significantly smaller particle sizes after being soluble in SCF. Drug particles can be micronized within a limited range of particle sizes, frequently to sub-micron levels, thanks to the flexibility and accuracy provided by SCF techniques.(8)

8) Solid Dispersion

One of the most popular techniques for improving the solubility of poorly soluble medications, primarily those of BCS classes II and IV, is the formation of solid dispersions. Solid dispersion is one of the most important strategies for dealing with the oral absorption of poorly soluble

compounds that is constrained by the rate of disintegration. Formulating weakly soluble compounds as solid dispersions may lead to improved wetting, decreased agglomeration, changed drug molecule physical state changeability, and possibly even a dispersion at the molecular level, depending on the solid dispersion's physical state. A group of solid goods composed of two or more separate parts; Solid dispersions typically consist of a hydrophilic matrix and a hydrophobic pharmaceutically active component. A solid can be either crystalline or amorphous. In the crystal lattice, the drug may be molecularly dispersed as clusters of crystalline or amorphous particles. One of the main factors influencing the drug's solubility in a solid dispersion formulation is this distribution.(19)

9) Inclusion Complexation

The inclusion complex creation technique has been used more specifically than any other solubility enhancement method to increase the aqueous solubility, rate of dissolution, and bioavailability of medications that are not very water soluble. Inserting a nonpolar molecule or the nonpolar portion of one molecule (referred to as the guest) into the cavity of another molecule or collection of molecules (referred to as the host) creates inclusion complexes. A tight fit between the guest and the host molecule's cavity is the main structural prerequisite for inclusion complexation. To minimize total contact between the water and the nonpolar areas of the host and the visitor, the host's cavity must be both big enough to fit the guest and tiny enough to remove water. Here is a list of the several methods used to get ready to create inclusion complexes of poorly soluble medications in an effort to increase their aqueous solubility.(8)

10) Self Micro Emulsify

The kind and quality of the surfactant concentration, the oil/surfactant combination, the oil/surfactant ratio, and the physiological conditions—such as pH and temperature—all affect self-emulsification. SEDDSs differ from traditional oral drug delivery systems in that the excipients in the formulation are drastically altered by the breakdown of enzymes (Amara et al., 2019).

Additional amphiphilic lipid digestion products are released in the GIT when the lipids in the oil phase of SEDDSs are hydrolysed by gastric and pancreatic lipases. Biliary lipids secreted in the bile quickly dissolve, and these released lipids are digested. During lipid digestion, the gastrointestinal lipolysis process is associated with many factors. These criteria include the secretions of pancreatic and gastric lipase, the pH differential between the stomach and small intestine, the pH of the lipase action, and bile secretions that enable micelle solubilization by lipolysis products. Over the years, SEDDS have also been developed to deliver hydrophilic macromolecular drugs, such as p DHA, peptides, proteins, and polysaccharides, orally.(20)

11) Liquisolid Technique

Both absorption and adsorption occur when the drug dissolved in the liquid vehicle is incorporated into a carrier material with a porous surface and closely matted fibers inside, such as cellulose. This means that the liquid is first absorbed in the particles' interior and is then captured by their internal structure, and once this process is saturated, the liquid is adsorbing onto the porous carrier particles' internal and external surfaces. The desired flow characteristics of the liquisolid system are then provided by the coating material's large specific surface area and high adsorptive qualities. Powdered forms of liquid medicines that

flow and compress well are known as liquidsolid solid systems.

In the concept of a liquisolid solid system, liquid drugs with low aqueous solubility are dissolved in appropriate non-volatile solvents and then transformed into a free-flowing, radially compressible powder by a simple admixture with specific powdered excipients known as carrier and coating materials. Microcrystalline and amorphous cellulose and silica powders may be used as coating materials. Liquisolid solid systems are acceptable flowing and compressible powdered forms of liquid medications.(8)

12) Microwave Irradiation Method

This technique uses a microwave oven, as the name implies, which results in a microwave irradiation reaction between the complex agent 30–34 and the medication. The medication and cyclodextrin are combined in a specific molar ratio in R.B.F. using an organic solvent and water solution. After that, the reaction is started in a microwave set to 60ºC 35–37 for one to two minutes. After the reaction is finished, enough solution is added. in order to eliminate the remaining, uncomplexed free medication and cyclodextrin. The Whatman filter is then used. paper, the precipitate is filtered, and it is dried for 48 hours at 40ºC in a vacuum oven.(21)

13) Microwave Induced Solid Dispersion Method

This approach uses a microwave to create the drug-polymer combination. The medicine and polymer fuse to form a solid dispersion when the mixture is exposed to microwave radiation, which raises the temperature in every component of the mixture. Solid dispersions created by microwaves seem to be a superior strategy to increase drug solubility compared to alternative techniques, since they are more practical and easier to prepare. There are several ways to disperse solids, including kneading, solvent evaporation, melting, hot melt extrusion, supercritical extraction, and microwave-induced fusion. The latter has several advantages over the others. When treating materials, the application of microwaves (MW) offers alluring benefits. Two groups of mechanisms—ion migration and dipole molecule rotation—cause MW heating, which results from the energy exchange between the electromagnetic (EM) field and the dielectric system (electro thermal coupling).(22)

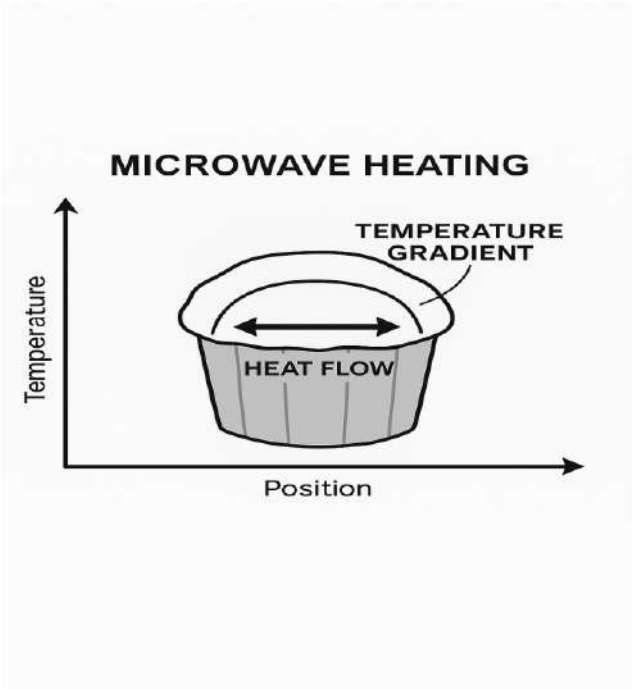

Figure 1 : Heat fluxes and temperature gradients produced in materials by microwave heating(22)

Advantages of Microwave Assisted Technique:

In the synthesis of inorganic nanomaterials, microwave radiation has shown itself to be a very efficient heating source. It provides the following benefits;

1) Effective heating source (superheating) that increases reaction rates and speeds up synthesis.

2) easy control over size and shape through adjustment of instrumental or reaction parameters.
3) selective heating based on the idea that different materials react differently to microwaves.
4) higher reproducibility of chemical reactions than with conventional heating due to uniform heating and improved control over process parameters.
5) potential pairings of microwave chemistry with other well-established liquid-phase synthesis routes, such as solvothermal and Sono chemical methods.(8)
6) The usage of microwaves results in a significant reduction in processing time.
7) It generates consistent warmth throughout the system.
8) When employing microwaves, a lower diffusion activation energy is needed.
9) It also aids in increasing the rates of chemical reactions. Its sintering temperature is lower.(22)
10) Microwave radiation is quickly emerging as a biological energy source. The high temperatures involved in microwave-assisted transformations are probably the reason for its delayed commencement as compared to organic synthesis.(23)

Applications of Microwave-Assisted Technique in Inorganic Nanomaterial Synthesis:

1) Microwave-assisted techniques have been widely applied in the synthesis of various inorganic nanomaterials due to their ability to provide rapid, uniform, and energy-efficient heating. They are particularly effective in the synthesis of metal oxide nanoparticles such as TiO_2, ZnO, and Fe_3O_4, which are used in photocatalysis, UV protection, and biomedical imaging, respectively. The technique enables precise control over particle size and crystallinity in significantly reduced reaction times.(24)

2) Microwave irradiation also facilitates the preparation of quantum dots like CdSe and ZnS with enhanced luminescence and narrow size distributions, making them suitable for applications in bioimaging and optoelectronics. Similarly, magnetic nanoparticles, especially Fe_3O_4, can be synthesized rapidly with uniform morphology for use in magnetic hyperthermia and targeted drug delivery.(25)

3) The method has proven useful in producing metal nanoparticles such as Ag, Au, and Pd for applications in catalysis, antimicrobial coatings, and sensors. Perovskite nanomaterials (e.g., $BaTiO_3$) and zeolites (e.g., ZSM-5) synthesized via microwave methods exhibit improved dielectric and catalytic properties due to faster crystallization and better phase control.(26)

4) Furthermore, hydroxyapatite nanoparticles prepared using microwave techniques are widely used in biomedical fields like bone repair due to their high purity and biocompatibility. Core–shell nanostructures, such as Fe_3O_4@SiO_2, benefit from microwave methods that ensure controlled shell formation and enhanced stability.(27)

5) An eco-friendly extension of this technique involves the green synthesis of nanoparticles using natural plant extracts under microwave conditions, which minimizes the use of harmful chemicals and reduces processing time—particularly useful in the development of antimicrobial agents and biosafe materials.(28)

CONCLUSION

Microwave-assisted solubility enhancement has proven to be a rapid, energy-efficient, and highly controllable approach for improving the dissolution rate and bioavailability of poorly water-soluble drugs. By leveraging uniform volumetric heating and targeted molecular interactions, microwave irradiation facilitates amorphization, particle size reduction, and formation of solid dispersions or inclusion complexes without the extensive use of organic solvents or prolonged processing times. Compared with conventional fusion and solvent-based methods, microwave-induced techniques minimize drug degradation, simplify workflow, and offer precise control over process parameters, making them particularly attractive for scalable pharmaceutical manufacturing.

Despite certain limitations such as the need for specialized equipment and careful optimization of dielectric properties these methods hold considerable promise for next-generation drug delivery systems and warrant further exploration in formulation design and industrial applications.

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