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POROUS MICROPARTICLES AS A VERSATILE PLATFORM FOR MODERN DRUG DELIVERY: A REVIEW

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ABSTRACT

Porous microparticles have gained significant importance in pharmaceutical formulation due to their ability to enhance solubility, bioavailability, and overall therapeutic performance of drugs with challenging physicochemical properties. Their high surface area, adjustable pore size, and interconnected pore networks enable efficient drug loading and improved dissolution behaviour. A wide range of preparation methods, including spray drying, freeze drying, anti-solvent techniques, electro spraying, and ultrasonic spray pyrolysis, provide flexibility in designing particles with tailored porosity and morphology. Drug loading strategies such as solvent evaporation, vacuum-assisted penetration, high-pressure methods, and supercritical fluid techniques further influence drug distribution and release patterns. Characterization approaches like SEM, FT-IR, DSC, XRD, and dissolution studies play an essential role in understanding particle structure and performance. Continued study into material selection, process refinement, and characterisation is required to fully realize their promise in current pharmaceutical development. Altogether, porous microparticles offer a versatile platform capable of delivering immediate, controlled, or sustained release profiles, making them a valuable advancement for developing effective and reliable drug delivery systems.

KEYWORDS: *Porous microparticles, Drug loading, Characterization, Preparation methods.*

INTRODUCTION

Poor bioavailability and drug dosage homogeneity, particularly in low-dose solid-drug products, are major obstacles to the design of oral dosage forms.[1,2]

The adsorption and deposition of active substances are made possible by the high pore volumes and increased surface area of porous materials. Porous materials are appealing for medication delivery because of these important features. To manage the rate of medication release, drugs are loaded into the porous structures.[3,4] In an effort to improve medication delivery, dissolving, tableting, and other processes, porous particles based on antiquated pharmaceutical materials were created as a novel carrier.[5]

Porous microparticles were invented in the 20th century and have since been used in a variety of fields, most notably in the pharmaceutical industry. Porous particles are classified into three categories based on their pore size diameter, according to IUPAC (International Union of Pure and Applied Chemistry) nomenclature:

1. Microporous (less than 2 nm),
2. Mesoporous (between 2 to 50 nm)
3. Macroporous (greater than 50 nm).

The diameter of porous particles can be changed by the preparation circumstances, which have a significant influence on their properties. Any solid that has voids or spaces that are not occupied by the main framework of atoms that make up the solid's structure is considered a porous microparticle. One characteristic of porous microparticles is their porosity. The chemical content and pore geometry size of porous materials vary widely.[6]

PPs are more suited for usage as drug carriers than conventional particles due to a number of unique characteristics, including large surface area, high porosity, homogeneous and adjustable pore structure, and well-defined inner and outer surface qualities.[7]

Porosity

The word pore comes from the Greek word '*πορος*', which means passage. This illustrates how a pore functions as a conduct between a solid's internal and external surfaces, enabling material to enter, pass through, or exit the solid. Pores of the third type is the transport pore, which links different parts of the solid's external surface to the interior microporosity, and the blind pore, which is connected to the transport pores but does not lead to any other pore or surface. These holes and how they are distributed throughout the solid structure are collectively referred to as porosity.[8]

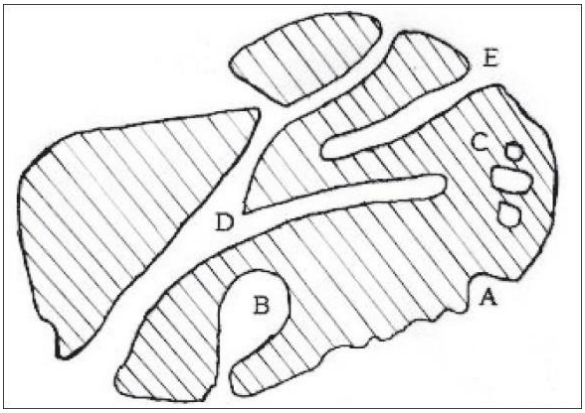

Fig. 1: Magnified physical picture of a porous microparticles physical picture of porous solid showing (a) - cylindrical blind, (b) - transport pores (pores through the solid), (c) - closed pores, (d) – surface roughness and (e) - ink bottle (blind pores).[8]

I. Microporosity

Micropores occur when crystallites are not aligned and small pseudo-graphitic crystallites form due to improper stacking and packing patterns in bulk materials. TEM investigations have shown that their shape is either slit-like or twisted. Adsorption in micropores can be totally reversed. Micropores come in three types: ultra micropores, micropores, and supermicropores. The pore diameter is similar to the adsorbate molecule, and ultra microporosity (0.5 nm) is commonly responsible for activated diffusion. Microporosity (diameter 0.5-1.4 nm) quickly fills during adsorption, with the pore wall potentials overlapping. Super microporosity (1.4-2.0 nm) promotes cooperative pore filling by forming monolayers and reducing pore width. This increases adsorption potential and completes pore filling at low relative pressure.[7,9,10]

II. Mesoporosity

Mesopores are caused by significant flaws in the micropores with a transport system by acting as passage within a solids structural framework. These pores are the origin of the capillary condensation phenomenon. The mesopores fill by multilayer formation. According to IUPAC definition, the pore widths are more than 2 nm but less than 50 nm, which means that under low relative pressures monolayer doesn't form. The adsorbed film serves as a nucleus upon which capillary condensation may occur. Coverage is followed by additional layers.[11]

III. Macroporosity

Micropores play a vital role in adsorption, while macropores primarily serve as transport pores. Solid structural faults such as breaks, splits, cracks, and etching grooves create macropores, similar to an open surface. Macroporosity can be detected using an optical microscope or scanning electron microscope on objects with a diameter of 50 nm or larger. The pores' diameter has no definite upper limit, but it is usually 1-2 nm.[12]

Porogen or templating agents

A common process for generating porous structures involves a pore generator, called porogen. A porogen template is a structured porous material or a porogen pattern whose geometry is inversely transferred to a second material. these processes generally named as porogen templating processes.[13] A porogen material can be a gas, a liquid, or an organic or inorganic solid. In a typical process, a porogen is mixed with a polymer solution or melt and after solidification is removed by solvent extraction, by evaporation or by other methods dependent on the type of the porogen and its phase state. Examples are salt leaching, melt blending, thermally or chemically induced phase separation, freeze drying, melt foaming, and solid-state foaming.[14]

A complete process for porogen templating contains three sequential stages,

(a) Preparing a porogen template with desired pore morphology including pore size, shape, connectivity, and distributions and gradients;

(b) Molding or casting of the desired polymeric material into the porogen template; and

(c) Removing the porogen template by dissolution, melting, or other processes from the molded part.

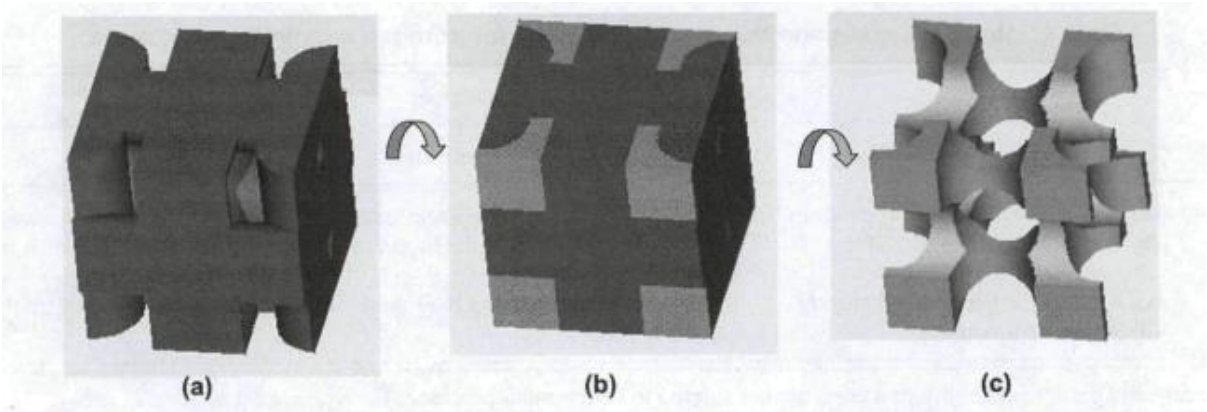

Fig. 2: A simplified illustration of the porogen templating process involving three sequential stages: (a) preparing a porogen tempiate; (b) molding or casting of the desired polymeric material; and (c) removing the porogen template.[14]

The porogen is usually an unreactive chemical that is added to the materials in order to create porous microparticles. The method by which porogens function is known as anti-solvent casting or particulate leaching. The pores on the outside and interior are formed by the spaces that the porogen particles leave behind after removal. Nevertheless, the process that requires the later extraction of the porogen causes other problems. The intrinsic benefits of the porogens pore-forming process include the following limitations: Antisolvent leaching encourages diffusional mass exchanges, which remove porogens and encapsulating actives. Furthermore, the high shear inherent in emulsification, which produces microparticles with wide dispersion in size and morphology, may be exacerbated by the presence of these porogen. Furthermore, porogens usually take some time to completely drain out.[15] Although there are other ways to prepare porous microparticles, co-processing the material with a porogen, such as ammonium bicarbonate, camphor, menthol, thymol, etc. is the most popular technique.[16]

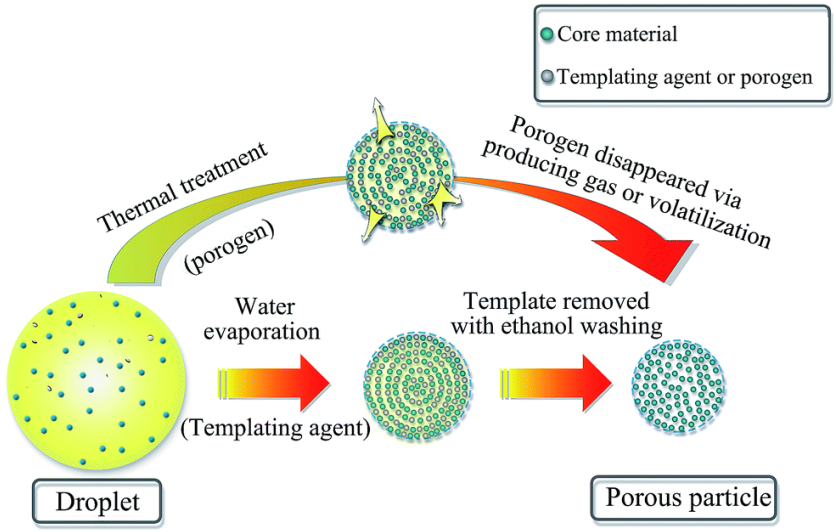

Fig. 3: The preparation of porous particles by using porogen or templating agent.[17]

MASS TRANSPORT IN POROUS MATERIALS[18]

All material loading and release processes in porous media are governed by mass transport, which is a general term for the movement of matter from one point to another. In order to examine and emphasize the impact of the loading technique and the selection of an appropriate carrier on the loading efficiency and drug release rate, we must first outline the basic principles behind mass transport across porous media. As a result, the primary features of porous structures and associated phenomena are discussed in the section that follows. Advection, diffusion, and electrophoresis are the three primary types of mass transport based on the driving forces at action. The different types of mass transport are given below.

I. Advection, bulk-flow, viscous flow[18–20]

Advection or bulk- or viscous flow can be defined as the transport of matter by a bulk motion of fluids, whereas the fluid can carry the transported material of interest, or it can be the fluid itself. The majority of the fluid is subject to cohesive and adhesive components, pressure, gravity, and other (mechanical) factors that induce fluid

motion or flow. Subcategories of these advective flows include –

a) Film Flow
b) Capillary flow
c) Permeation

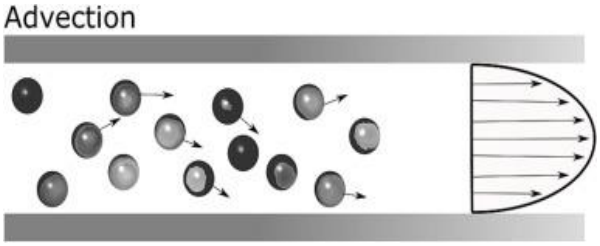

Fig.4: Schematic representation of the advective flow. Advective flow is defined as the transport of matter by a motion of the fluid bulk caused by a mechanical force.[21]

II. Diffusion-based mass transport

The transfer of materials from an area of higher concentration to one of lower concentration is referred to as diffusion. Brownian motion is what propels this movement, which results in a uniform distribution of atoms, molecules, ions, and tiny particles throughout the fluid's bulk. Systems based on dissolved matter in a liquid, like water or other solvents, are the focus of this section. The most pertinent diffusion phenomena for drug loading and release into and out of porous systems.

There are three types of diffusive motion in fluid-filled pores.

a) Continuum diffusion – It is the mass transport due to the Brownian motion.
b) Knudsen diffusion - describes the mass transport due to the collision of the molecules with the pore walls in materials with pore diameters smaller than the free path length of the molecules.
c) Surface diffusion - describes the movement of adsorbed species along the pore wall.

POROUS MICROPARTICLES PREPARATION TECHNIQUES

The various methods of preparation of porous microparticles are:

I. Anti-solvent method.
II. Freeze drying method
III. Ultrasonic spray pyrolysis method
IV. Using electrospray equipment
V. Spray-drying technique

These preparation methods fall into three categories:

A. Spray techniques.
B. Evaporation method.
C. Electrochemical etching.[22]

One method for creating porous microparticles is emulsion or suspension polymerization, in which monomer droplets are polymerized and the porogen is removed to create pores. Among the processes used to create porous microparticles are spray drying, freezing drying, electro-spraying, ultrasonic spray pyrolysis, and anti-solvent.[23]

Typically, emulsion polymerization takes place in oil-in-water (o/w) emulsions where hydrophobic monomers form the dispersed “internal” phase, usually between 10 and 30% of the emulsion. The monomers are emulsified within water (the continuous “external” phase) by using a water-soluble surfactant and polymerized by using a water-soluble radical initiator. A simplified description has the polymerization beginning with monomer molecules that have been solubilized within surfactant micelles through initiation at the oil−water interface. Monomer-swollen polymer nanoparticles (NPs), which usually have ultimate diameters between tens and hundreds of nanometres, continue to undergo polymerization. Hydrophilic polymer NPs can also be produced via emulsion polymerization utilizing "inverse" w/o emulsions. Microencapsulation, in which hydrophobic liquids are contained within micrometre-scale particles, is the second "emulsion" + "polymer" relationship that typically springs to mind. Interfacial step-growth polymerizations including isocyanates in the internal phase are commonly used for microencapsulation within o/w emulsions.[13]

1. Anti-solvent method

The porous microparticles are made by dissolving lactose in a solvent. After that, a porogen or templating agent was introduced to create porosity. The solvent solution is then mixed with an anti-solvent and left to precipitate lactose. After centrifugation, the slurry will be dried to a consistent weight.[24]

2. Freeze drying method

There is a report on the controlled freezing of emulsion to create porous microparticles. By freeze-drying the emulsion droplets, the oil and water solvent phases are removed, and the droplets are immediately frozen into solid microparticles. Because the frozen microparticles are contained in a supporting water-soluble polymer matrix that stays fixed until it is eliminated by subsequent dissolution, there are no worries about microparticle aggregation during solvent removal. The microparticles' porosity can be altered by only altering the internal emulsion phase's concentration or the freeze-drying process. More importantly, the emulsion can be directionally frozen to produce distinct porosity particles by creating a significant temperature difference between the emulsion droplets.[25]

3. Ultrasonic spray pyrolysis method

The ultrasonic spray pyrolysis method is a straightforward way for creating aerosols. A home humidifier's base has an ultrasonic transducer that provides the energy needed for liquid atomization. Precursor breakdown and solvent evaporation occur in a furnace that receives a fine mist of precursor droplets (diameters less than 5µm) carried by a gas. After being

gathered in bubblers at the end of the furnace, byproducts are either dissolved in the collection media or removed from the system by carrier gas. Using this technique, fine powders, microparticles, and nanoparticles have been produced. Additionally, precursor drops can be applied on heated surfaces to create films.[26]

4. Using electrospray equipment

The basic electrospraying system consists of a syringe pump, a metal nozzle connected to a high voltage power source, a grounded substrate serving as a collector, and a monitor. To enhance particle formation control and encourage the creation of smaller particles with better surface morphology, the electrospraying system should be isolated in a covered chamber. Electrospraying is used to form microparticles by injecting a stream of conductive liquid into a nozzle and applying a high voltage (KV range) to the droplets. This static charge generates an electrostatic force in the droplets. Only pieces of solution are jetted at low voltages, leading to unstable cone-jet modes, rapid dripping, and dripping. Stable cone-jet, multi-jet, and irregular unstable jet modes are produced by the equilibrium of many forces on the liquid surface, such as surface tension, gravity, and electric strengths. Surface tension, viscosity, electrical conductivity, and density are examples of liquid factors that affect the electrospray process.[27,28]

5. Spray-drying technique

PVP K30 was selected as the templating agent. The samples were made by varying the aqueous solution's templating ingredient while maintaining a constant lactose concentration. Clear solutions were spray-dried after all solutions were magnetically stirred for at least half an hour at room temperature (25 °C). A portion of the recently spray-dried powder was combined with sufficient alcohol to eliminate PVP K30, and the mixture was magnetically stirred for a full day at room temperature. Porous lactose was separated from alcohol containing PVP K30 using centrifugal force, and it was then dried for six hours at 45 °C in a vacuum chamber.[29]

TYPES OF POROUS PARTICLES

Table No. 1: Types of porous particles.

Si.No	Types	Key properties	Preparations	Functions and applications
1	**Porous starch (PS)**	High internal surface with granular, spherical shape. pore size ~1µm; total volume ~50% of starch	A freezing solvent exchange techniques or enzyme treatment	Solubility enhancement: Carbamazepine, normally water-insoluble, dissolves completely within 20 minutes when loaded into porous starch due to increased surface area and improved wetting. Stability: Drug retention and stability improve significantly, showing a 15–50% increase compared to conventional carriers.[30]
2	**Porous lactose (PL)**	The porosity and pore size can be adjusted.	Produced using the templating approach through spray drying	PL may increase the dissolving rate of poorly watersoluble medicines and make drug dosing more consistent. For example, 85% of the acetaminophen loaded in PL might be released within the first 5 minutes.[31]
3	**Porous mannitol**	The porosity and pore size can be tuned	Spray drying of mannitol solutions with porogen or templating agents	Dissolution: Within the first fifteen minutes, 80% of the nifedipine and indomethacin were dissolved. When cyclosporine A was attempted for pulmonary usage, its release increased nine times in twenty minutes.[32–34]
4	**Porous silicon**	The porosity and pores size can be adjusted.	Si is dissolved electrochemically in a solution based on hydrofluoric acid (HF).	Dissolution: when loaded to Si, the time for 80% ibuprofen release increased thrice, improving pharmacological activity.[22]
5	**Porous silica**	Sylysia 350: pore size, 21.0 nm.	Using emulsion template technique.	Dissolution: release amount of tanshinone II, increased ~4- times (at 180 min). Mucosal and systemic immune response improved.[35]
6	**Mesoporous silica nanoparticles**	Pore size, 2 to 50 nm	Reacting a micelle of surfactant and self-assembling silica	Dissolution: the amount of aceclofenac released increased about double (after two hours). Beneficial

			tetraethyl orthosilicate or sodium silicate.	for cancer treatment: improved effectiveness and fewer adverse effects.[36]
7	**Porous calcium silicate**	Porous structure, assembled petal-like flakes	Using reactivity-enhanced SiO2 as the silica materials at a low hydrothermal temperature	Dissolution: time for 80% meloxicam release is decreased by 30 folds.[37] Sustained release: release time of gentamicin, more than 5 days
8	**Porous ceramics**	Comprised of a three-dimensional array of hollow polygons	Using porogen or templating agent.	Achievement of sustained drug release[38,39]
9	**Functionalized calcium carbonate**	Size, 5–15 mm; a specific lamellar surface area, 40– 80 $m^2 g^{-1}$; porosity~70%	Re-precipitation of calcium phosphate incorporating calcium carbonate under controlled conditions	Tabletability: tablet tensile strength increases.[40]
10	**Porous chitosan**		Microemulsion combined with thermally induced phase separation technique	Hemostatic effect: hemostatic time is reduced by 1.5 folds.[41]

METHODS AND MECHANISMS OF LOADING

Using porous materials for drug delivery usually involves loading into the porous carriers. Loading the active pharmaceutical ingredient in the porous structure of the carriers is a prerequisite for taking advantage of these carriers' porosity and high surface area. That is in contrast to simple admixing or surface deposition. The rate at which the external payload reaches an equilibrium concentration inside the porous structure depends on the conditions of the loading process, the pore geometry, and surface characteristics.[42,43]

The results of drug loading in a porous carrier determine the drug release kinetics from that carrier. Understanding the physical and chemical mechanisms involved in deposition inside the porous material is necessary to control the drug loading process.[44,45]

Every loading procedure in a porous carrier includes an impregnation step, as previously mentioned. In this stage, the medication enters the porous structure either as a melted sample or with the aid of a solvent. A subsequent drying step is essential when a solvent facilitates infiltration. The distribution of the payload within the porous carrier is determined by the infiltration and drying processes. Because of the drug's interaction with the pore wall surface, the drug distribution using a certain loading method may differ from the anticipated result. This effect is pronounced in mesoporous materials and loading methods based on surface monolayer adsorption. Physical or chemical interactions result in the payload's adsorption on the pore walls.[46–48]

a) Simple mixing

This process involves adding the adsorbent to the medication solution and using a magnetic stirrer to whirl it for the appropriate time of duration. After an hour of standing, the solution is separated and dried for 24hrs at 60°. Among the medications that employ this method are furosemide, ranitidine, griseofulvin, dexamethasone, and ibuprofen.[15]

b) Solvent evaporation

Particle size variations are eliminated by sieving the adsorbent in the 250–350 µm range. After the drug was added to the solvent, the adsorbent was gradually added and left to evaporate at room temperature.[7]

c) Loading under high pressure

The medication was combined with the adsorbent in an adequate ratio and left in the high-pressure adsorption apparatus for 24 hrs. To get rid of any unentrapped medication, the powder was rinsed with deionized water and then dried for 5 hrs at 65° in a vacuum oven. This is how Brilliant Blue gets loaded.[34]

d) Vaccum process

In this method after adding the adsorbent to the medication solution, the mixer was sucked for a while before the vacuum was released. The medication solution and adsorbent were then left to stand for an hour. Solids were then separated using filter paper and dried for a full day at 60°. Several medications, including sodium benzoate, benzoic acid, and diltiazem hydrochloride, are used to load an adsorbent. In an alternative method, the medication and adsorbent are mixed in an appropriate volatile solvent for six hours before being evaporated under low pressure. For three hours, the resulting powder was vacuum-dried. Hydrophobic medications like phytonadione are loaded using this technique.[49]

e) Physical adsorption method

Physical absorption was used to put the medication into prepared porous lactose. In order to fully load porous lactose particles, the medication solution is pipetted onto them and allowed to sit for 30 minutes during physical absorption. The slurry was then centrifuged, and the supernatant was removed and checked for unloaded

medications. The residue was dried until its weight remained consistent.[27]

f) Fluidized bed dryer

This technique eliminates the need for repeated cycles of spraying and drying by allowing impregnation and drying to happen simultaneously. It involves three consecutive steps: 1) the drug solution in a suitable solvent is sprayed onto a porous carrier in a fluidized state, 2) the solution containing the active drug penetrates the porous carrier by capillary force, and 3) drying of the carrier loaded with the drug as they move around the bed. Controlling the spray rate, fluidization gas inlet temperature, and flow rate ultimately controls the impregnation process.[50]

g) Supercritical carbon dioxide technology (ScCO2)

Supercritical Approach Drugs are dissolved in supercritical fluids during impregnation. During the depressurizing stages, the drug precipitates from the critical solution as well as diffuses into the carrier matrix's pores. Supercritical fluids contain special characteristics such a density that is comparable to a liquid, viscosity that is comparable to gases, and increased diffusivity.CO2 is an inexpensive, harmless, and non-flammable fluid. The depressurizing procedure can readily remove it from the particles without leaving any solvent residue behind. CO2's low surface tension improves the impregnation process by keeping the medication from breaking down at a low working temperature.[51]

POROUS MICROPARTICLES CHARACTERIZATION

Table No. 2: Porous microparticles characterization.

Si.No	PARAMETERS	METHOD
1	Percentage yield (% yield)	It establishes how much polymer and medication are utilized in the preparation, as well as how many porous particles are produced in the end. The yield of porous particles can be computed as follows.[52] % Yield = Practical Yield /Theoretical Yield × 100
2	Particle size	It makes sure the formulation's particle size falls within the ideal range; particle size analysis is crucial. Particle size can be determined by optical microscopy.[52]
3	Carr's index & Hausner's ratio	The bulk density and tapped density were determined by a powder characteristics tester. The measuring cylinder was exactly 100 ml. The tapped time was 6 minutes for each formulation. The Carr's index & Hausner's ratio were calculated with help of following formula.[53] Carr's index = (Tap density – Bulk density) / Tap density × 100 Hausner' ratio = Tap density / Bulk density
4	Drug content estimation	Drug content analysis was carried out by dissolving the prepared formulations in an appropriate solvent and diluting them at the right concentration. Ultimately, a UV spectrophotometer was used to assess the drug content.[54] % drug Content = Practical drug content/ Theoretical drug content ×100
5	Drug entrapment efficiency	Entrapment efficiency can be determined by indirect method. In indirect method, free amount of drug was estimated in the supernatant and % entrapment efficiency was calculated using following formula.[52] % Entrapment Efficiency = $Theoretical\ Entrapment\ Practical\ Entrapment$ x100
6	Surface Morphology	It offers vital details about the microstructure and porosity of porous microparticles. The most used method for analyzing surface morphology is scanning electron microscopy (SEM). For this reason, the prepared sample is dehydrated because SEM needs a vacuum field to produce an image. Electron dense coating materials (such as gold, palladium, or a combination of both) are used to cover the samples (by sputter coating or thermal vacuum evaporation) prior to loading them for photomicrograph.[55]
7	Fourier Transform infrared Analysis (FT-IR)	To study the compatibility between the drug and polymer.[56]
8	X-ray Diffraction (X-RD)	X-ray diffraction was used to determine the formulations' structural patterns and crystallographic structure.[56]
9	Differential Scanning Calorimetry (DSC)	This method is utilized to gather both quantitative and qualitative data regarding the drug's physicochemical state within the porous microparticles. Endothermic or exothermic breakdown, outgassing, or a shift in the listed material's heat capacity are all included in DSC. This technique is used to track various samples of the same materials in order to examine their similarities and differences or the impact of additives on the formulation's thermal properties.[57]

10	In-vitro drug release studies of formulations	Dissolution apparatus was used for in-vitro drug release investigations, and the results were compared to pure drugs and commercial drug products.[52]
11	Stability studies of formulations	A stability study, which provides information on how the formulation's quality varies over time, was conducted on the optimized formulation. After the created formulations were kept in a stability chamber for a predetermined period of time at a predetermined temperature and relative humidity, changes in the formulation's physical characteristics and the amount of medication remaining could be assessed.[58]

CONCLUSION

Porous microparticles have emerged as a versatile and promising platform for enhancing drug delivery, owing to their large surface area, controllable pore structure, and ability to accommodate both hydrophilic and hydrophobic drugs. Their capacity to improve dissolution, stabilize sensitive molecules, and support controlled or sustained release makes them suitable for a wide range of therapeutic applications. Advances in templating techniques, carrier engineering, and loading mechanisms have enabled precise control over pore morphology and drug distribution, further strengthening their utility. As demonstrated across various porous materials, drug performance can be significantly improved through optimized design and processing strategies. Continued research into material selection, process refinement, and characterization will be essential to fully harness their potential in modern pharmaceutical development. Overall, porous microparticles represent a valuable and adaptable approach for overcoming challenges associated with solubility, stability, and targeted delivery in contemporary drug formulation.

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