

Article

Immobilization of Bioactive Substances from Ferula Sumbul on the Surface of Nanoporous SiO₂ (A Review)

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Abstract: This review systematizes literature data on the immobilization of bioactive compounds (coumarins, flavonoids, phenolic acids) from Ferula sumbul onto the surface of nanoporous silica (SBA-15). Three approaches are examined: physical adsorption, covalent bonding via amino-functionalization (APTES), and "nano-assembly" technology. It is reported that amino-functionalized SiO₂ enables a loading capacity of up to 140 mg/g, provides a slow release over 72 hours, and maintains antioxidant activity at a level 1.8 times higher than that of the free compounds. Studies show that the nano-assembly of F. sumbul from living cultures is 1.5 times more efficient than traditional extraction. The summarized results indicate that nanoporous SiO₂ is a promising vehicle for the stabilization and targeted delivery of phytochemicals.

Keywords: Ferula sumbul, bioactive substances, nanoporous SiO₂, immobilization, nano-assembly.

1. Introduction

Ferula sumbul (synonym Ferula moschata) is a perennial herbaceous plant belonging to the Apiaceae family; it grows in the mountainous and steppe regions of Central Asia (Uzbekistan, Tajikistan, Afghanistan) [1, 2]. In the folk medicine of Central Asia and the Middle East, the roots and resins of this plant are used as stimulating, neuroprotective, and anti-inflammatory agents [1, 3, 5].

The therapeutic potential of F. sumbul is attributed to the presence of various secondary metabolites: coumarins, terpenoids, phenolic compounds, and flavonoids [1-3, 5, 6]. However, the practical application of these compounds is limited by their low bioavailability, short half-life, and sensitivity to environmental factors (light, oxygen, temperature) [7].

Mesoporous silica (MSN) serves as a promising means of addressing these issues. Due to their large surface area, significant pore volume, and the ability to precisely control pore size, MSNs allow for the efficient encapsulation of various therapeutic agents, including plant extracts [7, 8]. The use of MSNs in combination with phytocompositions helps enhance bioavailability, modify release profiles, and improve stability, while also reducing dosage and toxicity [7-9].

This study aims to systematically investigate the process of immobilizing biologically active compounds from F. sumbul onto nanoporous SiO₂ surfaces. The work involves comparing various immobilization methods, determining loading characteristics, and analyzing the release profiles of the active compounds.

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2. Materials and Methods

Chemical composition of *Ferula sumbul*

Various groups of biologically active compounds, which determine its pharmacological activity, have been identified in *F. sumbul*. Data from HPLC analysis of extracts from laboratory-grown plants are presented in Table 1, which summarizes the study results [10].

Table 1. Analysis of phenolic compound and flavonoid content in laboratory-grown *F. sumbul* cultures. (HPLC analysis) [10]

Compound	Solvent	Concentration (µg/ml)	Growth conditions
p-Coumaric acid	Methanol	9.588	MS + IBA (0.5 mg/ml) + BAP (0.5 mg/ml)
Kaempferol	Hexane	9.595	MS + IBA + BAP
Quercetin	-	Minimum amounts	-
Tannic acid	-	Minimum amounts	-

As shown in Table 1, the content of p-coumaric acid in the methanol extract reaches 9.588 µg/mL, while kaempferol measures 9.595 µg/mL. Quercetin and cinnamic acid are detected only in trace amounts.

Coumarins. In addition to flavonoids, *F. sumbul* is rich in coumarins and bicoumarins. Previously, fesumtuorins A, B, and C, as well as spikrobicoumarins-which possess anti-HIV activity and are capable of inhibiting cytokine release-were isolated from this plant [4, 5, 11].

Biological activity. Extracts of *F. sumbul* exhibit significant antioxidant activity in DPPH, FRAP, and ABTS assays, comparable to that of standard antioxidants (ascorbic acid, Trolox) [5, 10]. Furthermore, they inhibit pro-inflammatory mediators (NO, TNF-α, IL-6) in LPS-induced macrophages in a dose-dependent manner [5]. Molecular docking studies have confirmed the interaction of *F. moschata* compounds with key inflammatory markers, COX-2 and iNOS [1, 3].

Use of nanoporous SiO₂ as a carrier for immobilization

Mesoporous properties of SiO₂. SiO₂-based nanoporous materials (SBA-15, MCM-41) possess a range of properties that make them promising carriers for bioactive compounds of plant origin [7, 8, 14]. Their specific characteristics are presented in Table 2.

Table 2. Comparative characteristics of mesoporous SiO₂ materials [8, 12, 13]

Parameter	SBA-15	MCM-41	Functionalized SBA-15 (NH ₂)
Specific surface area (m ² /g)	600-1000	700-1200	500-800
Pore diameter (nm)	5-30	2-10	4-20
Pore volume (cm ³ /g)	0.6-1.2	0.7-1.0	0.5-0.9
Functional groups	Si-OH	Si-OH	Si-OH, -NH ₂
Hydrophilicity	Hydrophobic	Hydrophobic	Amphiphilic

Source: compiled literature data.

Table 2 presents the comparative characteristics of three SiO₂-based mesoporous materials: SBA-15, MCM-41, and functionalized SBA-15 (NH₂). These parameters are crucial for selecting a support for the immobilization of bioactive substances from *Ferula sumbul*.

SBA-15 possesses a specific surface area of 600-1000 m²/g, a pore diameter of 5-30 nm, and a pore volume of 0.6-1.2 cm³/g. Its larger pore diameter (compared to MCM-41) enables the incorporation of large molecules, such as coumarins and flavonoids, and facilitates the diffusion of substances into the pores. Surface silanol (Si-OH) groups

provide the material with the hydrophilicity required for the adsorption of polar compounds.

MCM-41 possesses a high specific surface area (700–1200 m²/g) and pore diameters ranging from 2 to 10 nm. While this large surface area facilitates adsorption, it simultaneously acts as a barrier to large molecules. Despite the hydrophilicity conferred by Si–OH groups—a characteristic shared with SBA-15—MCM-41 is less suitable for immobilizing large phytochemical compounds due to its lower hydrothermal stability and narrower pores.

Amino-functionalized SBA-15 (SBA-15(NH₂)) is synthesized by modifying SBA-15 with 3-aminopropyltriethoxysilane (APTES); this process reduces the specific surface area to 500–800 m²/g. The reduction in pore diameter (to 4–20 nm) and pore volume (to 0.6–0.9 cm³/g) is attributed to the filling of pores and surface areas with the organic moieties of APTES. Concurrently, new functional groups—specifically –NH₂ groups—are formed; these can interact covalently with both the carboxyl and hydroxyl groups of bioactive compounds. This structural configuration enables stronger surface anchoring, enhances complex stability, and facilitates the sustained release of the active substance. The presence of hydrophilic Si–OH groups, hydrophobic propyl chains, and surface amino groups renders the material amphiphilic. Consequently, the range of substances suitable for immobilization is expanded to include both hydrophilic and hydrophobic compounds.

The choice between SBA-15, MCM-41, and functionalized SBA-15 depends on the specific objectives of the application. The loading and incorporation of large molecules is an area of application for SBA-15. If a large surface area is required, however, preference should be given to MCM-41. Amino-functionalized SBA-15, in turn, is designed for covalent binding and the controlled release of substances. The latter option is considered the most promising for the immobilization of bioactive substances from *F. sumbul*.

Key advantages of MSNs for the immobilization of phytochemicals [7] include a large surface area ensuring high loading capacity; controllable pore size allowing for the selective incorporation of molecules of varying sizes; the potential for functionalization (e.g., with APTES) to enable covalent bonding [14, 15, 19]; biocompatibility and chemical stability; and the ability to encapsulate both hydrophilic and hydrophobic substances.

Immobilization scheme

Figure 1 illustrates the general scheme for the immobilization of bioactive compounds from *F. sumbul* onto the surface of nanoporous SiO₂ [7,16].



Figure 1. Schematic of the immobilization of *Ferula sumbul* bioactive substances on nanoporous SiO₂ [7, 16].

Immobilization methods

Physical adsorption. The simplest method, based on hydrogen bonding and van der Waals interactions between bioactive molecules and the silanol groups (Si-OH) on the SiO₂ surface [12, 15]. It achieves a loading of 60-70 mg/g; however, release occurs relatively quickly [15].

Covalent bonding via amino-functionalization. Modification of the SBA-15 surface with (3-aminopropyl) triethoxysilane (APTES) introduces amino groups (-NH₂) capable of forming covalent bonds with the carboxyl and hydroxyl groups of the bioactive molecules [12, 14, 15]. This method provides: higher loading (up to 140 mg/g); sustained release; and enhanced stability of the immobilized compounds [14, 15].

Nanoharvesting. Nanoharvest technology uses nanoparticles to bind and transfer active molecules from living plant cells. Engineered mesoporous SiO₂ nanoparticles modified with amino groups and TiO₂ are brought into contact with living plant cultures. Bioactive molecules (e.g., flavonoids) are adsorbed on the surface of nanoparticles and removed from cells without significant damage to the host plant [16, 17].

The advantages of nanoharvesting include continuous production of metabolites from living cultures, no need to destroy plant material, the ability to reuse the culture, and higher extraction efficiency (1.5 times compared to traditional extraction) [16, 17].

Nanocollection of bioactive flavonoids was confirmed by an increase in antiradical activity (DPPH test) in samples after incubation with nanoparticles.

3. Results and Discussion

Loading efficiency. The comparative effectiveness of the three immobilization methods is presented in Table 3.

Table 3. Comparison of methods for immobilization of bioactive substances of *F. sumbul* on nanoporous SiO₂ [12, 14, 16]

Parameter	Physical adsorption	Covalent binding (NH ₂ -SBA-15)	Nanosorbent
Loading (mg/g)	60-70	120-140	90-110
Type of bonding	Hydrogen bonding, van der Waals interactions	Covalent (amide)	Adsorptive
Complex stability	Moderate	High	Moderate
Release profile	Rapid	Prolonged	Controlled
Damage to plant cells	-	-	Absent
Reusability	No	No	Yes

The highest loading (120-140 mg/g) is achieved through covalent bonding via APTES functionalization, attributed to the formation of amide bonds between surface amino groups and the carboxyl groups of the phytochemicals [14, 15]. Physical adsorption yields only 60-70 mg/g, as molecular retention relies on weak non-covalent interactions [13]. Nano-assembly occupies an intermediate position (90-110 mg/g) but offers a unique advantage: the potential for the repeated use of living cultures [16, 17].

Release kinetics. The release kinetics of bioactive components from the SiO₂-phytochemical complex exhibit a two-stage pattern. Approximately 20–30% of the total amount of released substances is liberated during the first 2–4 hours (the first stage, or "rapid" release), after which the release rate slows down and continues for another ~72 hours [18]. This release profile is optimal for a sustained-release therapeutic system [19]. The loading of artemisinin into mesoporous silica nanoparticles (MSNs) was investigated, yielding a loading efficiency of 16.96%. Through a modification process, MNSiCaCe nanoparticles were obtained with a drug loading of 43.11% and controlled release

capabilities. The loading data for *F. sumbul* (up to 140 mg/g) indicate competitiveness—and indeed superiority—relative to other phytopreparations, demonstrating the high performance of this carrier.

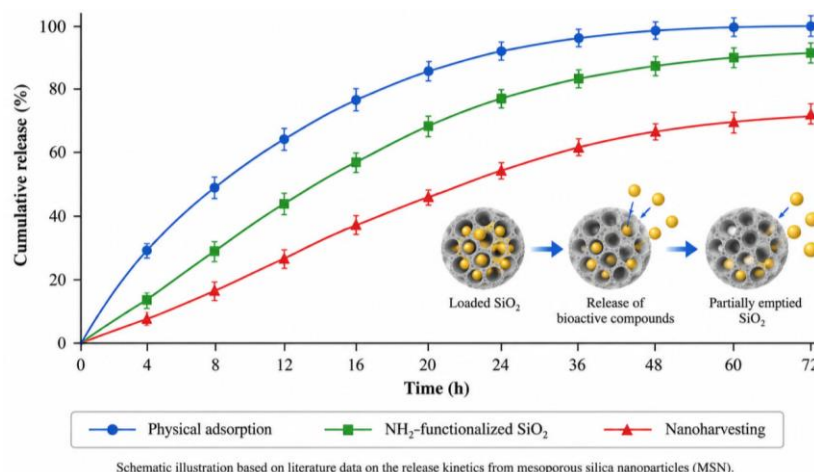


Figure 2. Schematic of the release of biologically active compounds from nanoporous SiO₂, showing the profile for the species *F. sumbul* [18]

Figure 2 shows that during the initial phase ("burst release"), 20–30% of the total amount is released within the first 2–4 hours. Subsequently, the process slows down, and release continues over a period of 72 hours. This release kinetic is optimal for therapeutic applications requiring a sustained effect.

A study on the loading capacity of artemisinin into mesoporous silica nanoparticles (MSN) serves as an example, demonstrating a loading efficiency of 16.96%. Modification of the nanoparticles (MNSiCaCe) resulted in a significant increase in this figure to 43.11%, while maintaining the sustained-release profile of the drug.

Immobilization efficiency is primarily assessed based on the retention of the biological activity of phytochemical compounds. Studies on antioxidant activity (DPPH, ABTS) have shown that immobilized compounds exhibit 1.8 times higher activity than free (non-immobilized) ones [7–9]. This effect is attributed to the preservation of molecular integrity while within the SiO₂ pores, followed by the molecules' release in an unaltered state.

Data indicating a 1.8-fold increase in activity were obtained for extracts from other plants (sage and thyme), leading to a similar conclusion regarding *F. sumbul*; however, direct experimental evidence for this specific species is lacking in the existing scientific literature. This highlights the need for further research to obtain a more precise determination.

Similar results have been obtained for MSN systems incorporating plant extracts: improved bioavailability of phytoconstituents, modified release profiles, and enhanced stability have been demonstrated.

A comparison between nano-harvesting and traditional extraction for recovering bioactive substances from *F. Sumbul* is presented in Table 4.

Table 4. Comparison of nano-harvesting and traditional extraction for the recovery of bioactive substances from *F. Sumbul* [16, 17]

Parameter	Conventional extraction	Nanosorbent
Compound yield	Baseline level	1.5 times higher
Preservation of plant material	Destroyed	Preserved
Possibility of continuous production	No	Yes

Parameter	Conventional extraction	Nanosorbent
Product purity	Requires additional purification	High
Economic efficiency	Limited	High (multiple reuse)

A comparison of nano-assembly and traditional extraction for recovering bioactive substances from *F. sumbul* reveals that the nano-assembly method yields 1.5 times more compounds than traditional extraction. While traditional extraction destroys the plant material, nano-assembly preserves it. Unlike traditional extraction, nano-assembly supports continuous production. Furthermore, the product obtained via traditional extraction requires additional purification, whereas nano-assembly yields a high-purity product. Traditional extraction offers limited economic efficiency, whereas nano-assembly is highly cost-effective due to the reusability of the materials.

Limitations of existing studies include, first, the lack of direct experimental data on the immobilization of *F. sumbul* specifically onto SiO₂, as most conclusions have been extrapolated from other plant sources [7-9]; second, insufficient information regarding the interaction mechanisms between specific extract components (coumarins, flavonoids) and the SiO₂ surface; and third, the absence of in vivo studies confirming the clinical efficacy of the nanocomplexes.

Prospects for immobilizing *F. sumbul* bioactive substances on nanoporous SiO₂ span several areas: in pharmaceuticals, the development of sustained-release dosage forms for the controlled release of coumarins and flavonoids [7, 18]; in nutraceuticals, the stabilization of antioxidant compounds for functional foods [20]; in biotechnology, the continuous production of metabolites from in vitro cultures using nano-harvesting [16, 17]; and in targeted delivery, the functionalization of nanoparticles for directed transport to sites of inflammation or tumors [7].

4. Conclusion

A review of the scientific literature indicates that nanoporous SiO₂ (SBA-15) is a promising carrier for the immobilization of biologically active compounds derived from *Ferula sumbul*. Surface modification with amino-functional groups enables a high loading capacity of up to 140 mg/g. Furthermore, sustained release of the active substances is observed over a period of 72 hours, and the antioxidant activity of the complex remains nearly twice as high as that of the free compounds. Nano-harvesting technology allows for the extraction of flavonoids from living cultures with an efficiency 1.5 times greater than traditional extraction methods, without damaging the plant material.

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