Recycled Polyester Composite Fiber Structure with Inorganic Shell Powder Dispersed Phase

Abstract

This novel invention relates to a recycled polyester composite fiber structure with a shell powder dispersed phase. It is characterized by comprising a recycled polyethylene terephthalate (r-PET) fiber matrix with a special cross-section, containing 5–30 wt% of calcined natural shell powder particles (e.g., oyster shell powder, with a particle size of 2–8 μm). These particles are surface-modified to be uniformly and discontinuously dispersed within the fiber, with some exposed on the fiber surface to form a micron-scale roughness structure. Its cross-section has a Y-shaped, trilobal, or other special-shaped design, and can further include hollow micropores to enhance breathability and functional surface area. Through this structural design, the novel invention combines excellent deodorizing, antibacterial, moisture-absorbing, and spinnability properties, making it suitable for the development of functional and sustainable textile materials.

【Specified representative picture】 Figure 1

【Simple description of symbols representing the picture】

1- Regenerated polyethylene terephthalate
2- Renovated shell powder particles

Recycled Polyester Composite Fiber Structure with Inorganic Shell Powder Dispersed Phase

Specification

【Technical Field】

【0001】 The present invention relates to a composite fiber material, and more particularly to a recycled polyester-based composite fiber structure with a dispersed phase of calcined shell powder. The composite fiber is environmentally friendly and functional, and is suitable for use in the fields of functional textile products and sustainable materials.

【Prior Art】

【0002】 Traditional composite fiber production processes often use virgin polyester (Polyethylene Terephthalate, PET) blended with organic or inorganic fillers to improve fiber performance. However, the use of inorganic powders from natural sources (such as calcium carbonate and talc) often suffers from problems such as uneven dispersion, poor interface compatibility, and insufficient processing thermal stability. At the same time, increasingly stringent environmental protection requirements have led to a trend in developing fibers based on recycled materials. Although existing recycled polyester fibers have environmental advantages, they are still insufficient in mechanical strength and functionality (such as deodorization and moisture absorption). Dispersing organic or inorganic fillers in fibers can improve some properties, but there are problems such as poor dispersion or weak interface bonding. 【New Content】

【0003】 To address the above-mentioned technical problems, this novel invention proposes a recycled polyester composite fiber structure with a shell powder dispersed phase, the main features of which include:

1. Calcined shell powder (mainly composed of calcium oxide (CaO)) is used as an inorganic dispersed filler and surface-modified with a silane coupling agent to enhance its interfacial compatibility and bonding strength with recycled polyethylene terephthalate (PET).

2. Recycled polyethylene terephthalate (r-PET) is used as the main polymer matrix. The surface-modified shell powder is blended with recycled polyethylene terephthalate (r-PET) to form a masterbatch, which is then mixed with recycled polyethylene terephthalate (r-PET) raw material sheets and melt-spun to produce continuous filaments or staple fibers with specific geometric cross-sections.

3. The resulting fiber consists of discontinuously dispersed inorganic powders. Clear particle distribution and enrichment can be observed in cross-section and longitudinal sections. Some particles are exposed on the surface, creating a rough structure that enhances antibacterial, deodorizing, and moisture-absorbing properties.

4. The fiber is specially designed with Y-shaped and cross-shaped cross-sections, and incorporates a hollow porous structure to increase the fiber's specific surface area and air permeability. The pore diameter and particle size are in a designed ratio.

【0004】Compared to conventional recycled polyethylene terephthalate (r-PET) fibers or pure cellulose acetate (CA) fibers, this new fiber offers the following technical benefits:

1. Higher mechanical strength and better thermal stability;

2. Controllable surface roughness, facilitating post-processing hydrophilic modification;

3. Deodorizing and antibacterial properties;

4. Excellent spinnability, suitable for the development of functional and eco-friendly textiles;

5. Can be co-spun with other fibers to form composite functional materials.

【Brief Explanation of Figures】

【0005】

Figure 1. Schematic diagram of the structure of the novel composite fiber

Figure 2. Longitudinal cross-section of the novel fiber (showing the shell powder dispersed phase)

Figure 3. Cross-section of the novel masterbatch (showing the shell powder dispersed phase)

Figure 4. Comparison of masterbatch and fiber structure

Figure 5. 3D reconstruction of the shell powder dispersed phase

Figure 6. Production flow chart: shell powder modification, masterbatch preparation, and fiber melt spinning process

Figure 7. Scanning electron microscope image of modified shell powder

Figure 8. Scanning electron microscope microscopic distribution of the cross-section of the composite fiber

Figure 9. Thermal stability test of the control group (no shell powder) and the novel fiber

Figure 10. Performance comparison of the control group (no shell powder) and the novel fiber

Figure 11. Scanning electron microscope image simulation of the masterbatch and nearest neighbor distance analysis of the Weibull distribution of the novel fiber

Figure 12. Visualization of multi-level pores and specific surface area (BET) enhancement

Figure 13. Infrared spectrum (FTIR) simulation

Figure 14. Surface roughness-adsorption performance correlation curve

Figure 15. Schematic diagram of fiber structure with a special-shaped geometric cross-section

Figure 16. Performance test curve of this new fiber

【Implementation Method】

【0006】This novel structure is a recycled polyester composite fiber structure with a shell powder dispersed phase. Its core features include:

1. Compositional Characteristics of the Composite Fiber Structure:

The fiber is composed of a continuous phase matrix of recycled polyethylene terephthalate (r-PET) 1, with calcined natural shell powder 3 particles dispersed within the fiber structure as an inorganic dispersed phase (as shown in Figure 1). The shell powder has an average particle size of 2–8 μm and is dispersed in the r-PET matrix at a ratio of 5–30 wt%, with 25 wt% being preferred. The shell powder particles, after surface modification with a silane coupling agent (such as KH-550), form a stable interfacial bond with the polymer matrix (as shown in Figure 5), enhancing bonding strength and long-term stability. 2. Structural Design of Special-Shaped Fibers:

The fiber cross-section features a special geometric shape, such as a trilobal 18, a Y-shaped 19, or a hollow structure 20 (as shown in Figure 15). Porous regions can be optionally configured to increase specific surface area and adsorption performance. In the cross-section, the shell powder particles are observed to be discontinuously distributed, with some particles exposed on the fiber surface, forming a micron-scale roughness structure (as shown in Figure 2). The surface roughness Ra ranges from 0.5–2.5 µm (as shown in Figure 14), which helps enhance the fiber's gas and liquid adsorption performance.

3. Functional Interface and Microscopic Distribution:

The inorganic filler and the recycled polyethylene terephthalate (RPTE) matrix form a stable chemical interface bond (as shown in Figure 5), which not only enhances the overall mechanical properties of the fiber but also improves the uniformity of the powder dispersion. In some embodiments, the shell powder is locally enriched or distributed in a gradient pattern. This structure facilitates the formation of a functional layer on the fiber surface, enhancing the concentrated and effective antibacterial and deodorizing properties (as shown in Figure 10). This structure is stably achieved by controlling the masterbatch ratio and spinning parameters. As shown in Figure 11, the Weibull distribution shape parameter is 1.97 and the ratio is 4.84, indicating a concentrated distribution, good material consistency, and high overall strength.

4. Functional Performance (Figure 16):

Because the calcined shell powder3 is primarily composed of calcium oxide (CaO), it possesses excellent alkalinity and ion exchange capacity, imparting significant deodorizing and antimicrobial properties to the fiber.21 According to ASTM D6646 testing, the fiber's ammonia adsorption rate can reach 87%. Antimicrobial testing conducted in accordance with JIS L1902 demonstrates an antibacterial rate exceeding 95% against Staphylococcus aureus. Furthermore, in terms of mechanical properties23, the fiber exhibits excellent tensile strength (approximately 3.8 cN/dtex) and elongation (12%), making it suitable for general woven and nonwoven applications. 5. Optional Structure:

This novel fiber can also be further designed with hollow regions, where the hollow region accounts for 20–36% of the total cross-sectional area (as shown in Figure 15), and the pore diameter is less than half the average particle size of the shell powder used, thereby enhancing the fiber's lightweight and breathability. Furthermore, designs can also feature regions with localized particle concentrations, where the distance between particles is less than 1.5 times the average particle size, to form a surface functional layer or active interface.

【0007】 The production method, as shown in Figure 6, is as follows: calcined shell powder 3 is ground into shell powder with an average particle size of approximately 2-5 μm, and then surface-modified 4 with a silane coupling agent. The modified calcined shell powder is then mixed with recycled polyethylene terephthalate (r-PET) 1 at a weight ratio of 25:75 or 15:85 to form a functional masterbatch. The masterbatch is then blended 5 with recycled polyethylene terephthalate (r-PET) 1 sheet at a ratio of 10-30 wt%. The masterbatch is then produced into long or short fibers using a single-screw or twin-screw melt spinning 6 machine.

【Explanation of Symbols】

【0008】

1- Recycled polyethylene terephthalate

2- Modified calcined shell powder particles

3- Calcined shell powder

4- Surface modification

5- Masterbatch blending

6- Melt spinning

7- Control group

8- Novel fiber

9- Masterbatch

10- Percolation path

11- Data

12- Weibull distribution

13- Nanopore

14- Micropore

15- Hierarchical pores

16- Key peak

17- Correlation curve

18- Trilobal shape

19- Y-shape

20- Hollow structure

21- Deodorization performance

22- Antibacterial performance

23- Mechanical properties

24- Hollow pores

Claims

【Claim 1】 A recycled polyester composite fiber structure with a shell powder dispersed phase, comprising a continuous, hollow, irregularly shaped fiber with a cross-section, wherein the fiber comprises:

A recycled polyethylene terephthalate (r-PET) matrix, forming the continuous fiber phase and the irregularly shaped cross-section;

A calcined natural shell powder dispersed phase, having an average particle size of 2–8 µm, uniformly dispersed in the r-PET matrix at a ratio of 5–30 wt%, forming a discontinuous dispersed phase, as observed in the fiber cross-section;

The calcined natural shell powder is surface-modified with a silane coupling agent and is bonded to the recycled polyethylene terephthalate (r-PET) matrix. The fiber forms chemical bonds at the interface of the raw material and the raw material (r-PET) to enhance dispersion stability and interfacial adhesion;

The fiber cross-section has a well-defined geometric shape, such as a Y-shape, a trilobal shape, or a cross star shape, and has a hollow microporous structure. The irregular geometric structure and hollow design enhance the fiber's functionality;

Some of the calcined shell powder particles are exposed on the fiber surface, forming a micron-scale roughness structure (Ra 0.5–2.5 µm) with a Weibull distribution shape parameter of 1.2–2.0;

The fiber has a deodorizing function: an ammonia adsorption rate of ≥ 85% (ASTM D6646 standard test).

【Claim 2】 A regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the calcined natural shell powder dispersed phase has an average particle size of 2–6 µm, a content of 10–25 wt% of the total fiber weight, and a Weibull distribution size parameter of 1.5–3.0 µm (as determined by scanning electron microscopy image analysis, sample size ≥ 500 particles). 【Claim 3】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the profiled geometry is trilobal, hollow porous, or cross-shaped, and the shell powder concentration in the concave and convex areas of the profile is 10–20% higher than in the flat areas.

【Claim 4】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the calcined natural shell powder particles are distributed in a gradient pattern in the longitudinal direction of the fiber, and the particle number density within 0.5 μm from the fiber surface is 1.5–3 times that of the core.

【Claim 5】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the thickness of the interface transition layer between the shell powder and recycled polyethylene terephthalate (r-PET) is 50–200 nm (measured by transmission electron microscopy).

【Claim 6】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the fiber cross section comprises a hollow porous structure, wherein the hollow portion accounts for 15–35% of the total cross-sectional area, and the average pore diameter is less than half the average particle size of the calcined natural shell powder, thereby further increasing the specific surface area and air permeability.

【Claim 7】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the contact angle between the surface-modified calcined natural shell powder and the recycled polyethylene terephthalate (r-PET) matrix is less than 30° (demonstrating interfacial bonding strength).

【Claim 8】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the fiber surface roughness (Ra) is 0.5–2.5 μm, and the roughness peak corresponds to exposed shell powder particles (measured data range: $R^2 \geq 0.92$, n=20 samples).

【Claim 9】 A regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the calcined natural shell powder particles have an aspect ratio of 1.5–3.0 (melt stretching causes deformation).

【Claim 10】 A regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the single fiber tensile strength is 3.5–4.8 cN/dtex, and the difference is within ±10% compared to pure recycled polyethylene terephthalate (r-PET) fiber.

【Claim 11】 A regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the calcined natural shell powder dispersed phase includes a local enrichment region in which the interparticle spacing within the region is less than 1.5 times the average particle size, thereby improving the fiber surface roughness and functional distribution strength.

【Claim 12】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the nearest neighbor distance distribution of the calcined natural shell powder particles in the fiber cross section conforms to a Weibull distribution (shape parameter 1.2–2.0).

【Claim 13】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the crystal orientation of the calcined natural shell powder particles (measured by X-ray diffraction) has a deviation angle of less than 15° from the fiber axis.

【Claim 14】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, which is produced by blending a masterbatch containing 15–25 wt% calcined natural shell powder and recycled polyethylene terephthalate (r-PET) in a weight ratio of 10:90 to 30:70.

【Claim 15】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, which is suitable for use in moisture-wicking textiles or heavy metal adsorption filter materials.

【Claim 16】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein its 24-hour formaldehyde decomposition rate is ≥ 70% (JIS L 1902 test), attributed to the alkaline catalytic effect and porous structure of the shell powder.

【Claim 17】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the shell powder particles have a specific surface area of 5–15 m^2/g (measured by the BET method), and the pore diameter is 2–50 nm, forming a multi-level adsorption structure with the roughness peaks on the fiber surface.

【Claim 18】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein its antibacterial rate (against S. aureus) is ≥ 90% (AATCC 100), attributed to the sustained release of calcium ions from the shell powder and its rough surface structure.

【Claim 19】 The recycled polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the heavy metal adsorption capacity is:

Lead ion (Pb^{2+}) ≥35 mg/g;

Cadmium ion (Cd^{2+}) ≥28 mg/g;

(tested according to ISO 17294-2, pH=5, 25°C).

【Claim 20】 The recycled polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the fiber elongation at break is increased by 10–20% compared to pure recycled polyethylene terephthalate (r-PET) fiber, attributed to the crack deflection effect of the shell powder dispersed phase (in situ tensile observation using a scanning electron microscope).

【Claim 21】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein its infrared spectrum exhibits a characteristic peak at 1,540 cm^{-1} (evidence of interfacial bonding between shell powder calcium carbonate and regenerated polyethylene terephthalate ester groups).

【Claim 22】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein its X-ray diffraction analysis spectrum shows that the crystal plane orientation of the shell powder is ≥ 0.75 (calculated by the Herman orientation factor).

【Claim 23】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein dynamic mechanical analysis (DMA) shows that the storage modulus (E') decreases by less than 15% in the range of 30–80°C, indicating interfacial bonding stability.

【Claim 24】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the masterbatch preparation parameters include:

Twin-screw extruder temperature setting: 220–250°C (to avoid thermal decomposition of the shell powder);

Shear rate: 500-800 s^{-1} (to ensure that the Weibull distribution shape parameters of the dispersed phase meet the requirements).

【Claim 25】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, suitable for medical textiles, exhibiting a deodorizing function retention rate of ≥80% after five standard washes (AATCC 135 test conditions).

【Claim 26】 The regenerated polyester composite fiber structure with a shell powder dispersed phase as described in Claim 1, wherein the fiber diameter coefficient of variation (CV%) is ≤8% (n=100 measurements), reflecting the uniformity of the dispersed phase.

Figures

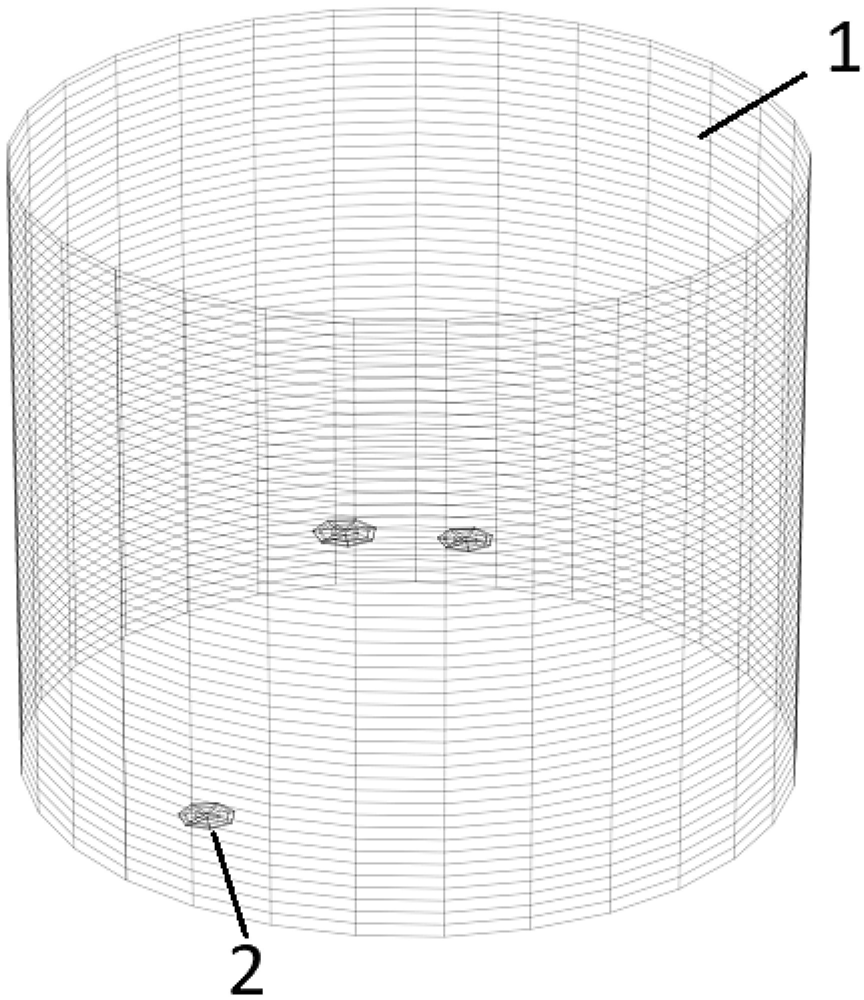

FIG 1.

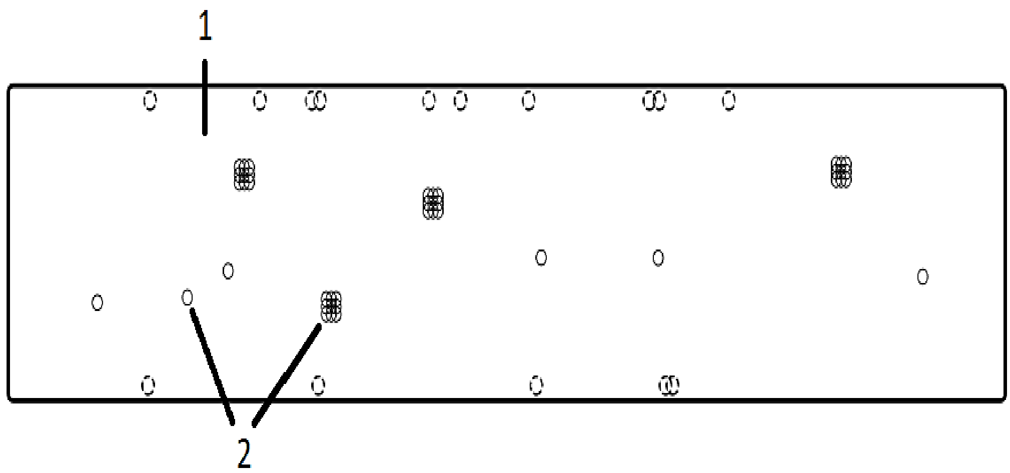

FIG 2.

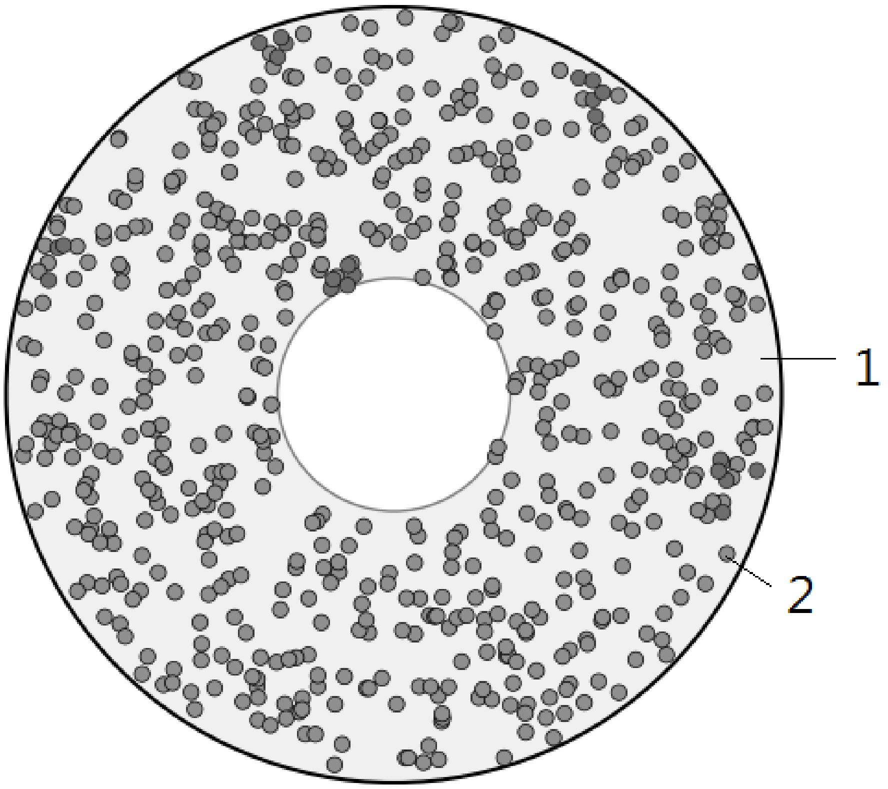

FIG 3.

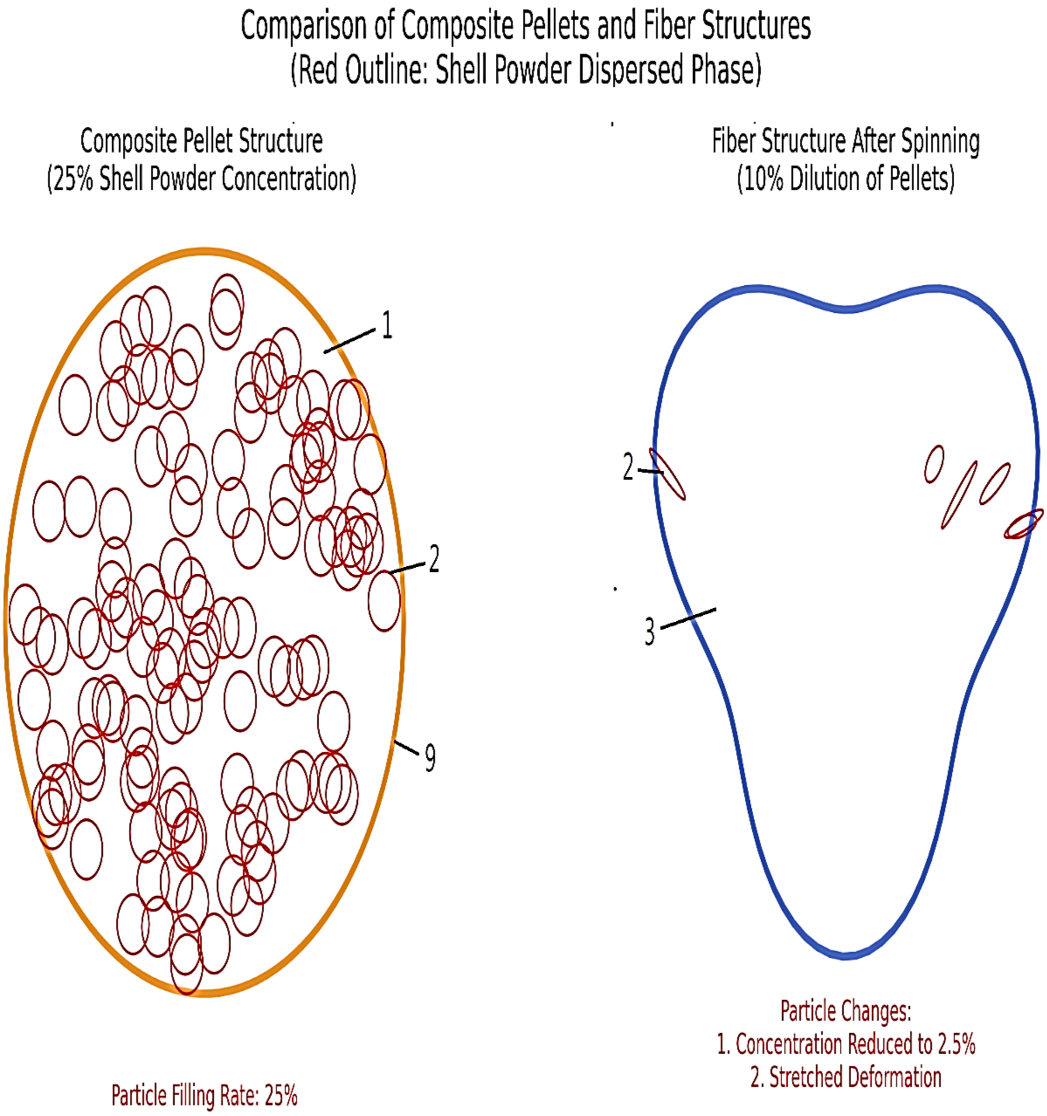
Comparison of Composite Pellets and Fiber Structures
(Red Outline: Shell Powder Dispersed Phase)
Composite Pellet Structure
(25% Shell Powder Concentration)
Fiber Structure After Spinning
(10% Dilution of Pellets)
1
2
9
2
3
Particle Filling Rate: 25%
Particle Changes:
1. Concentration Reduced to 2.5%
2. Stretched Deformation

FIG 4.

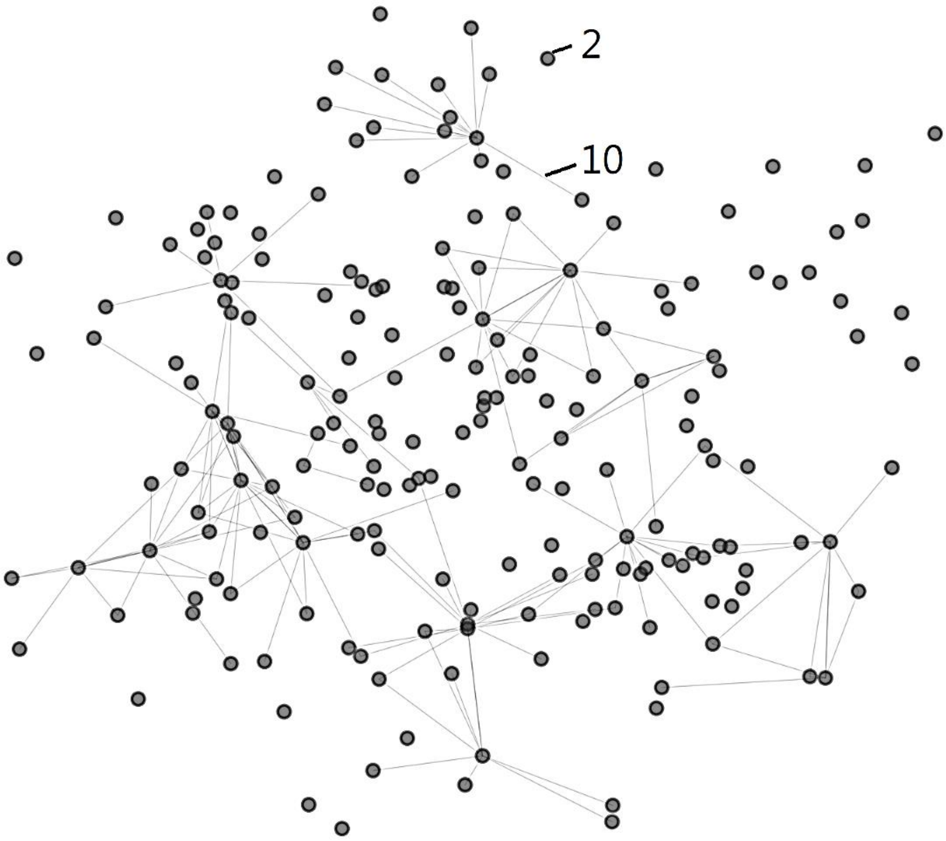

FIG 5.

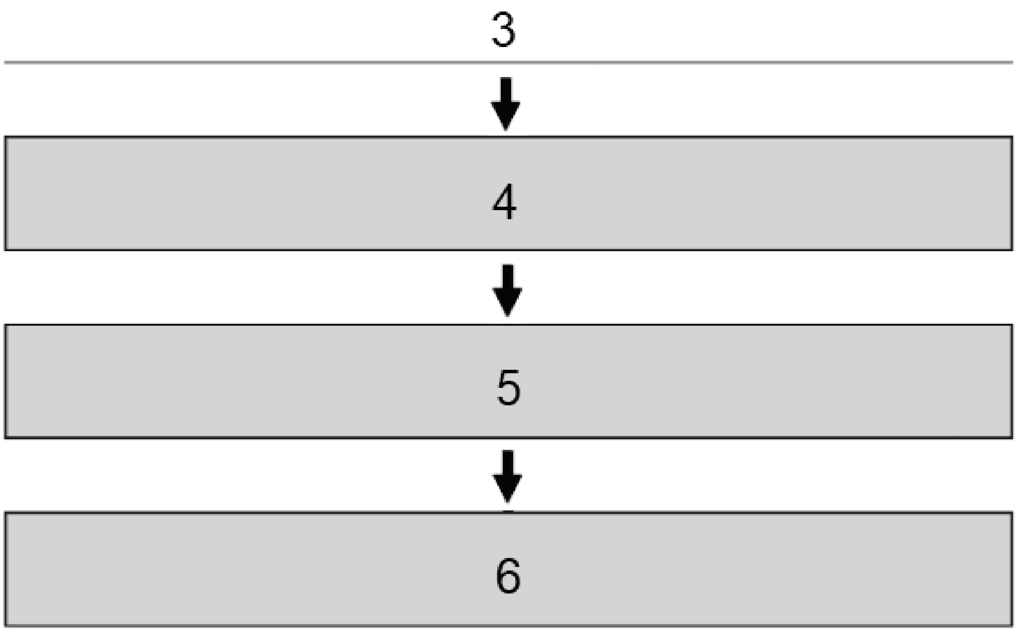

FIG 6.

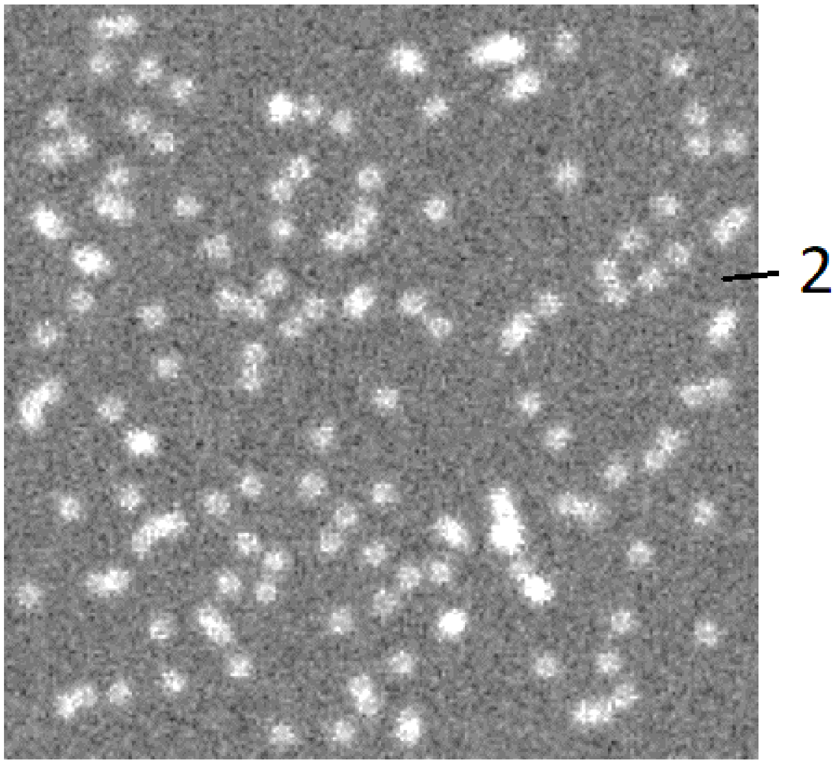

FIG 7.

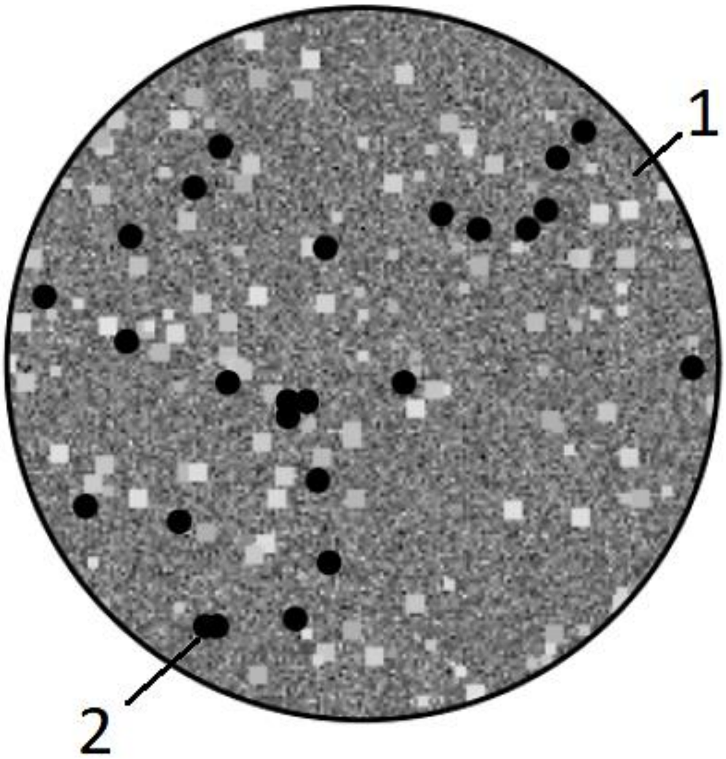

FIG 8.

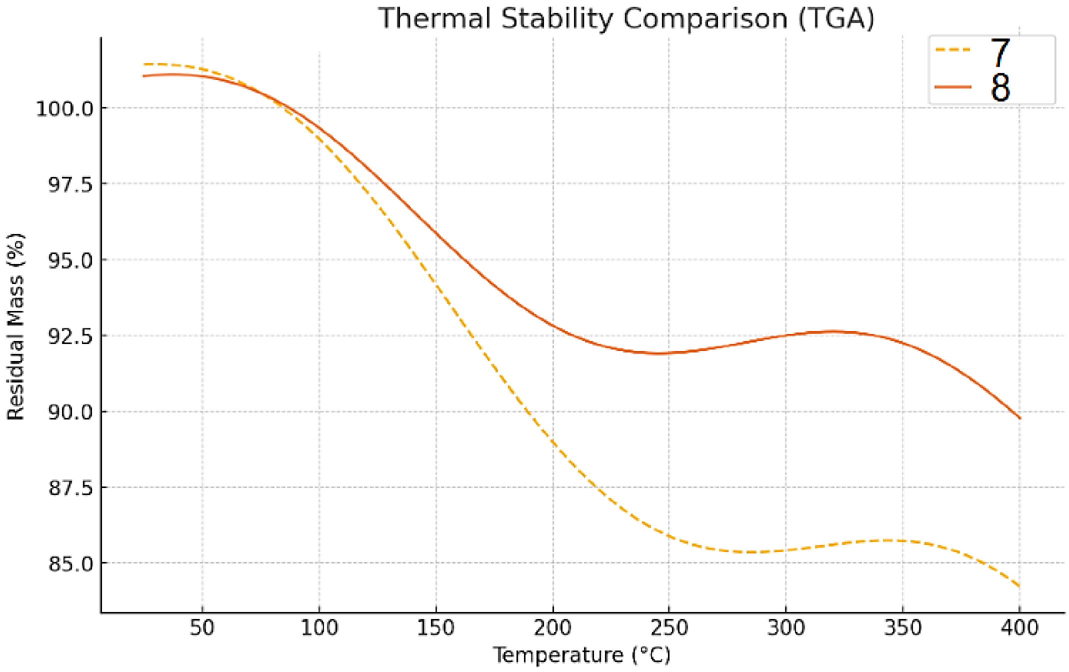
Thermal Stability Comparison (TGA)
7
8
Residual Mass (%)
100.0
97.5
95.0
92.5
90.0
87.5
85.0
50
100
150
200
250
300
350
400
Temperature (°C)

FIG 9.

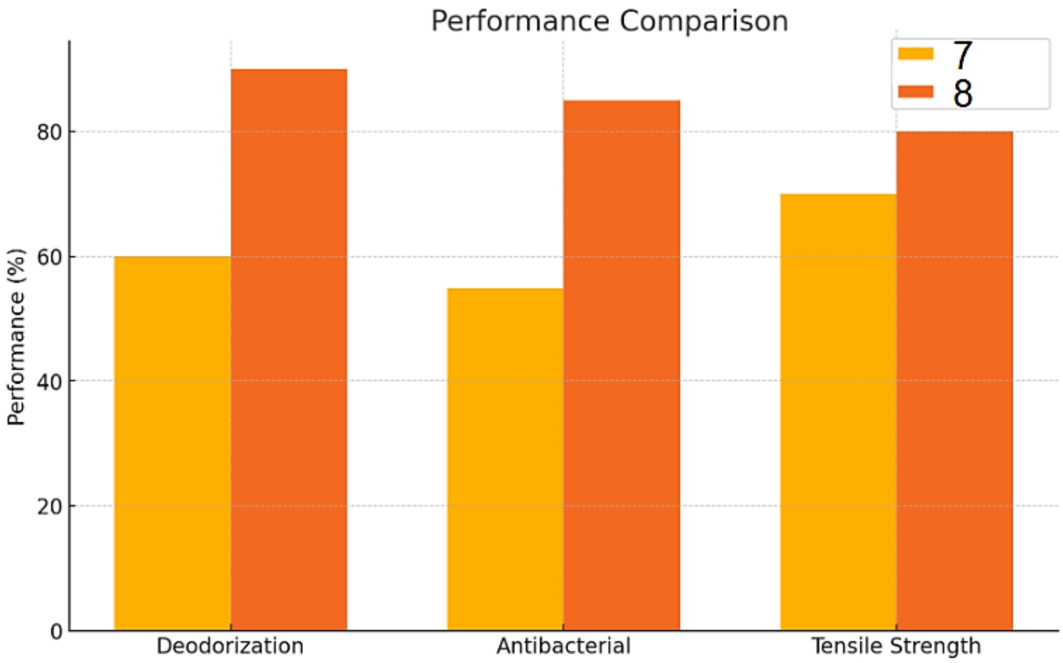
Performance Comparison
7
8
Performance (%)
80
60
40
20
0
Deodorization
Antibacterial
Tensile Strength

FIG 10.

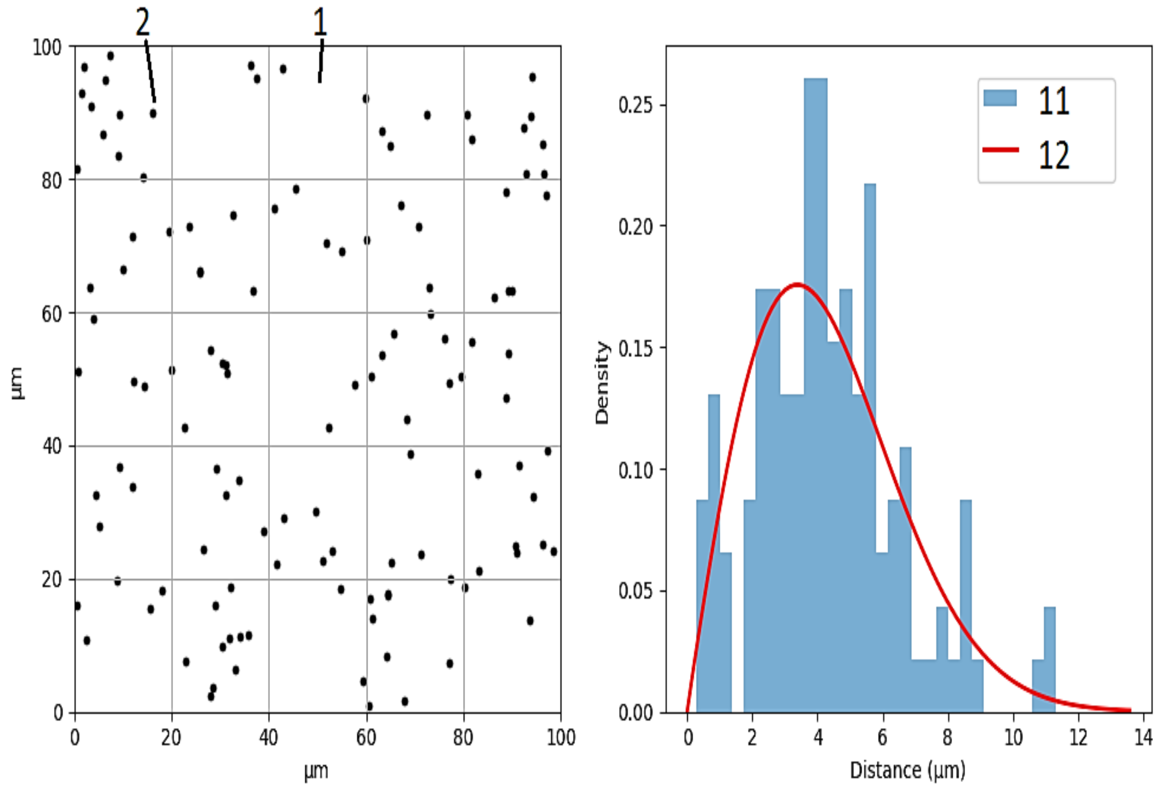
2
1
µm
µm
11
12
Density
Distance (µm)

FIG 11.

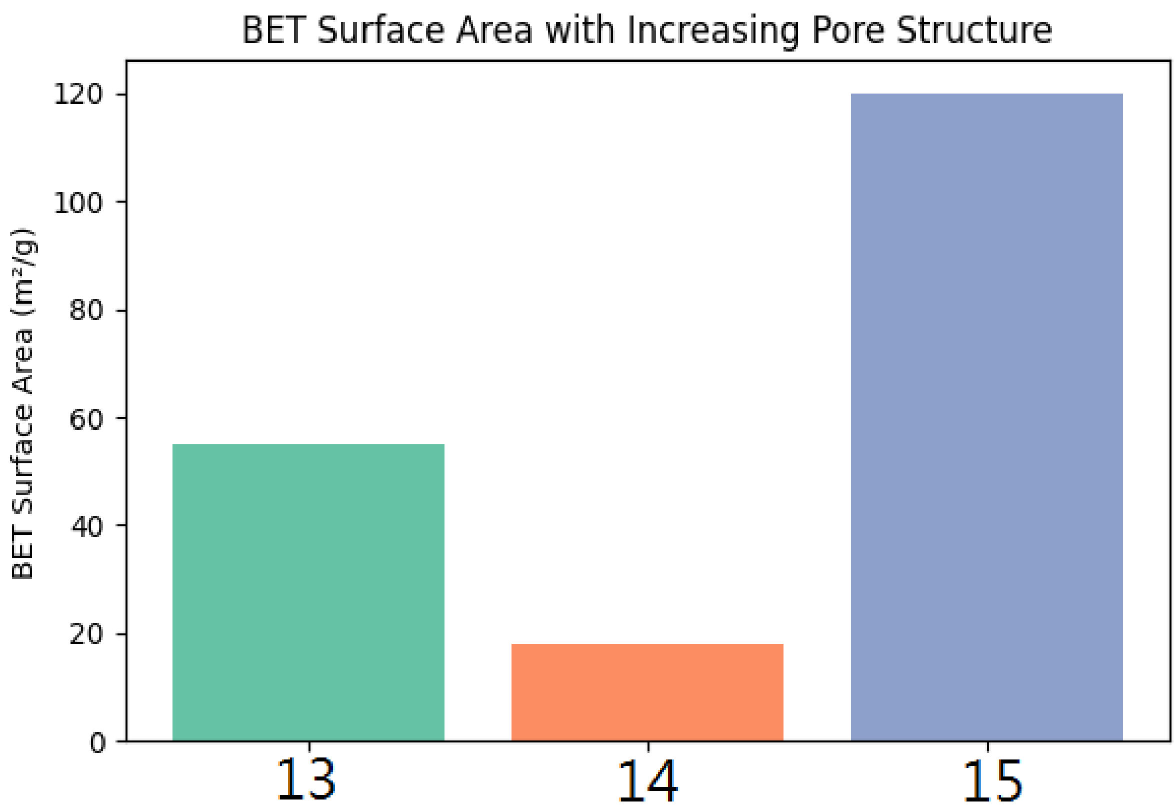
BET Surface Area with Increasing Pore Structure
BET Surface Area (m²/g)
13
14
15

FIG 12.

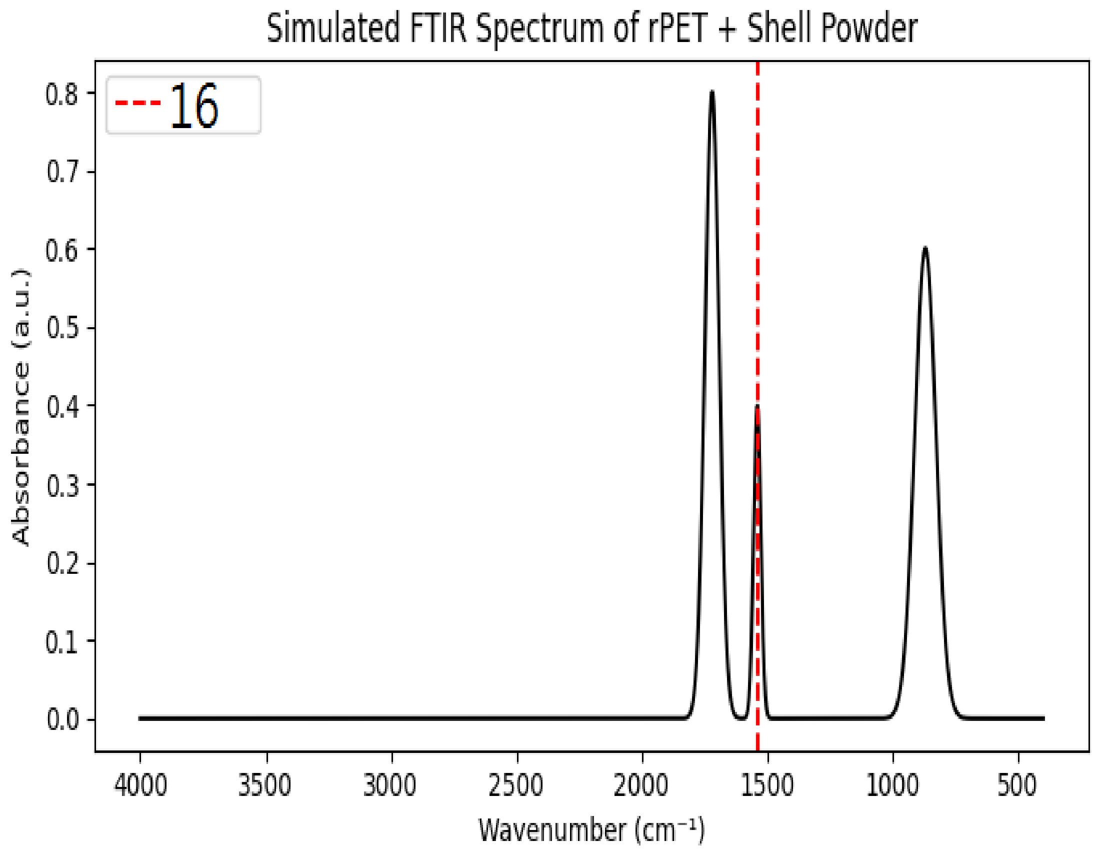
Simulated FTIR Spectrum of rPET + Shell Powder
16
0.8
0.7
0.6
0.5
0.4
0.3
0.2
0.1
0.0
Absorbance (a.u.)
4000
3500
3000
2500
2000
1500
1000
500
Wavenumber (cm⁻¹)

FIG 13.

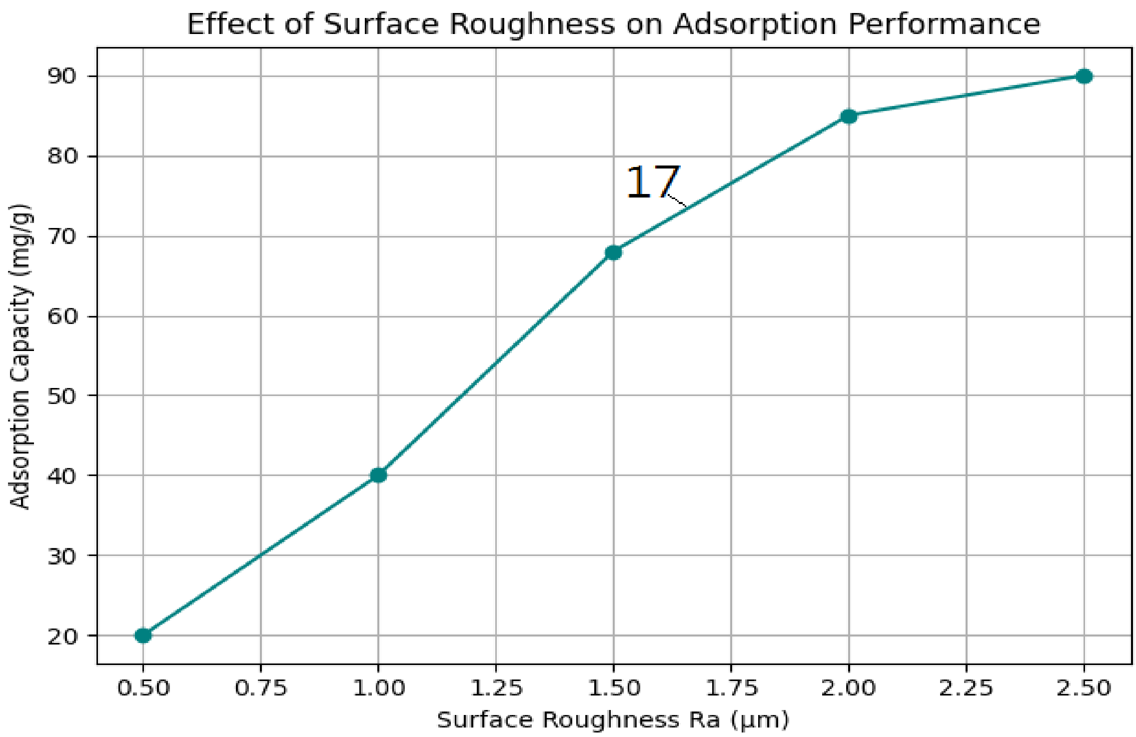
Effect of Surface Roughness on Adsorption Performance
17
90
80
70
60
50
40
30
20
Adsorption Capacity (mg/g)
0.50
0.75
1.00
1.25
1.50
1.75
2.00
2.25
2.50
Surface Roughness Ra (μm)

FIG 14.

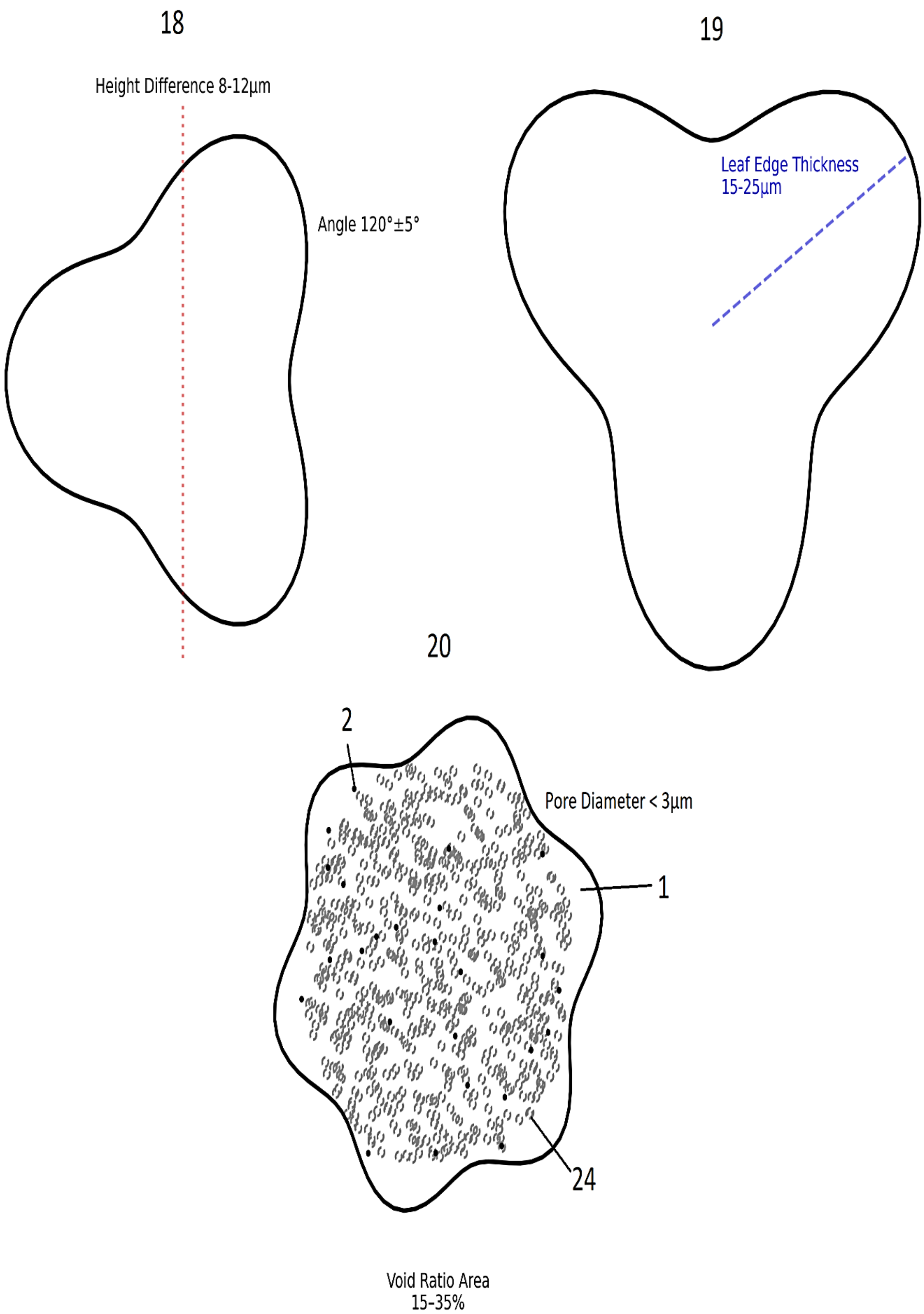
18
Height Difference 8-12μm
Angle 120°±5°
19
Leaf Edge Thickness
15-25μm
20
2
Pore Diameter < 3μm
1
24
Void Ratio Area
15-35%

FIG 15.

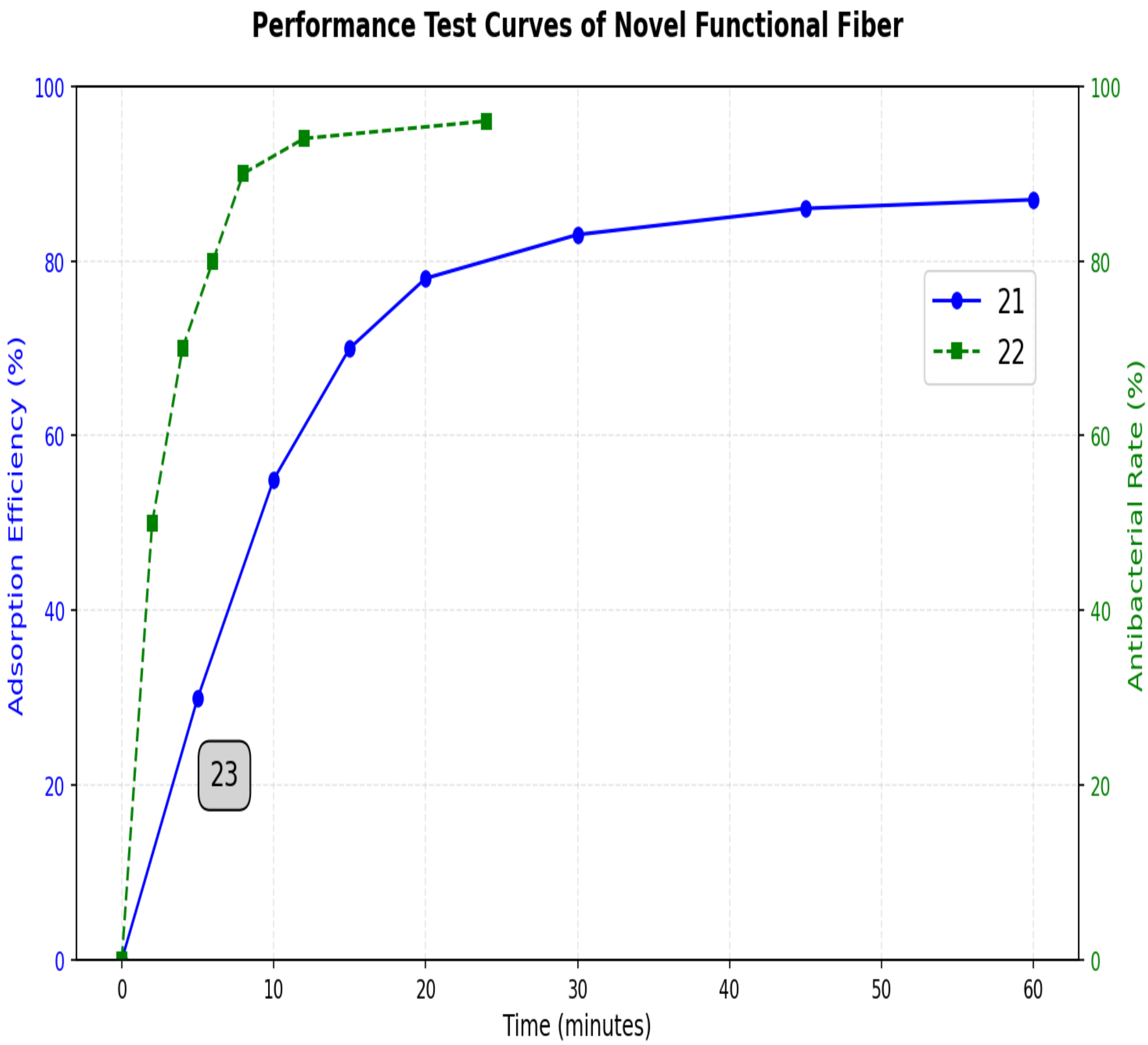
Performance Test Curves of Novel Functional Fiber
Adsorption Efficiency (%)
Antibacterial Rate (%)
Time (minutes)
0
10
20
30
40
50
60
80
100
21
22
23

FIG 16.