Electronic Supplementary Material (ESI) for ChemComm.

Supplementary information

High shear spheroidal topological fluid flow induced coating of polystyrene beads with C_{60} spicules

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Experimental

Materials and Chemicals

Fullerene C_{60} (99.5% purity) and poly(styrene-co-divinylbenzene) (6-10 μm specified average particle size, with the diameter determined herein using SEM as ca 2 – 6 μm) (99.5 purity) were suspended in toluene, with both materials and solvent purchased from Sigma Aldrich.

Sample preparation and Materials synthesis

Solutions of fullerene C_{60} were prepared in toluene at different concentrations, 0.1, 0.5, 1, and 2 mg.mL^{-1}. Initially, as-received fullerene was added to toluene, and the mixture was allowed to stand at room temperature for 24 h, whereupon it was filtered to remove undissolved particles before mixing with polystyrene beads dispersed in toluene in the VFD using different jet feeds, through Adelab new era NE-300 single syringe pumps in a 50 mL glass syringe. The operating parameter space for the VFD was systematically explored, namely the rotational speed of the rapidly rotating glass tube, tilt angle of the tube, type and size (OD = outside diameter) of tube and concentrations of polystyrene beads and fullerenes. After VFD processing, the resulting material was collected via centrifugation at g = 1.751 rfc for 30 min and the solid collected on a filter paper. Fullerene C_{60} material generated under the shear stress re-dissolves in toluene over several hours, but this allows sufficient time to collect the material with minimal re-dissolution post VFD processing.[1]

Characterization

Samples of C_{60} on silicon wafers were prepared by drop casting a colloidal suspension in toluene followed by evaporation under ambient conditions. The morphology, size, and shape of the particles and their properties were studied using a number of complementary techniques including scanning electron microscopy (SEM), using an accelerating voltage of 5 kV, operating at 10 mm working distance. X-ray diffraction (XRD) data was collected using a Bruker ADVANCE D8 ECO, Co Kα, at an operating wavelength of 1.7988 Å with 2θ varied from 10 to 90°. Raman spectra were recorded to study the nature of the products, using a Horiba XploRA apparatus at a fixed wavelength of 532 nm. UV-visible spectra were carried out to determine percentage of C_{60} in solution, using a Cary 50 and Aligent 60 UV- vis spectrometer. A blank of toluene was used for baseline analysis with a slow scan from 800 to 200 cm^{-1}. All experiments where run under these conditions. Thermogravermetric analysis (TGA) was determined using a Perkin Elmer STA 8000 instrument operating under a nitrogen atmosphere with a temperature range 30 to 800 ºC (30 to 700 ºC for coated beads) with a 10 ºC/min increase (approximate weight of samples was 2 to 4 mg).

Supplementary Data

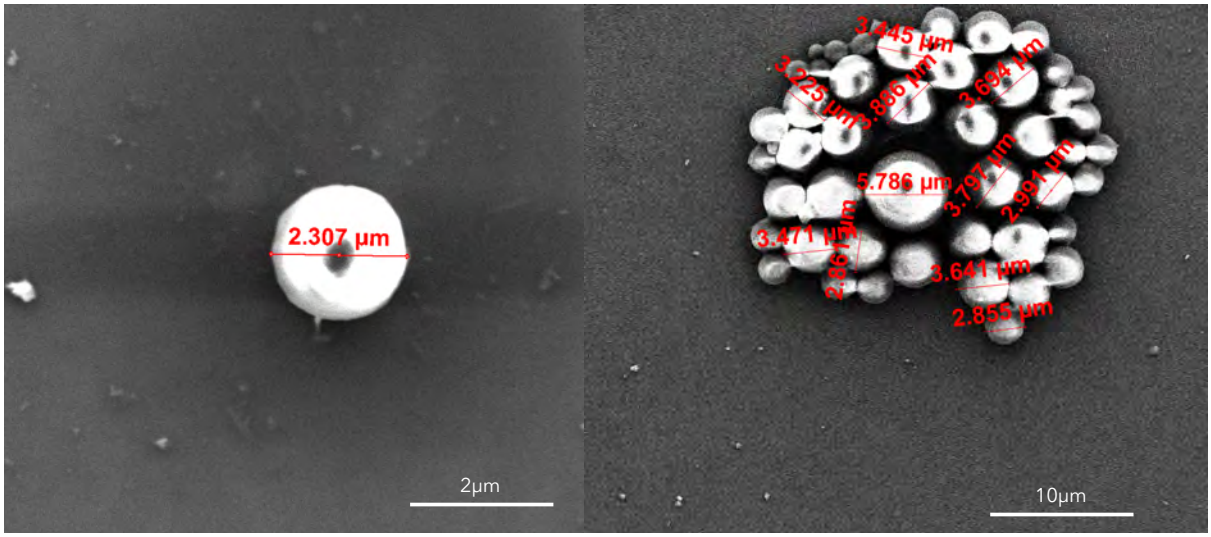

Figure S1: SEM Images of the poly (styrene-co-divinylbenzene) as received after dispersing in toluene, then drop casted on to a silicon wafer.

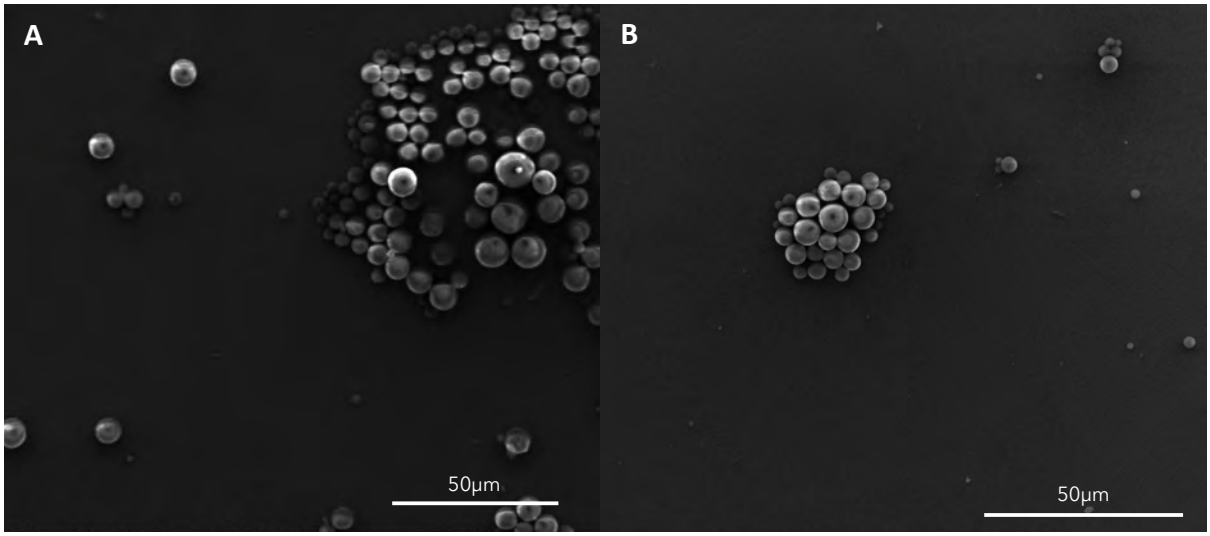

Figure S2: SEM Images of the poly(styrene-co-divinylbenzene) beads drop casted onto a silcon wafer, after 30 minutes processing in the VFD under the following conditions: rotational speed (A) = 4k rpm (B) 7.5k rpm, tilt angle (θ) = 45°, concentration 0.1 mg.mL^{-1} in toluene.

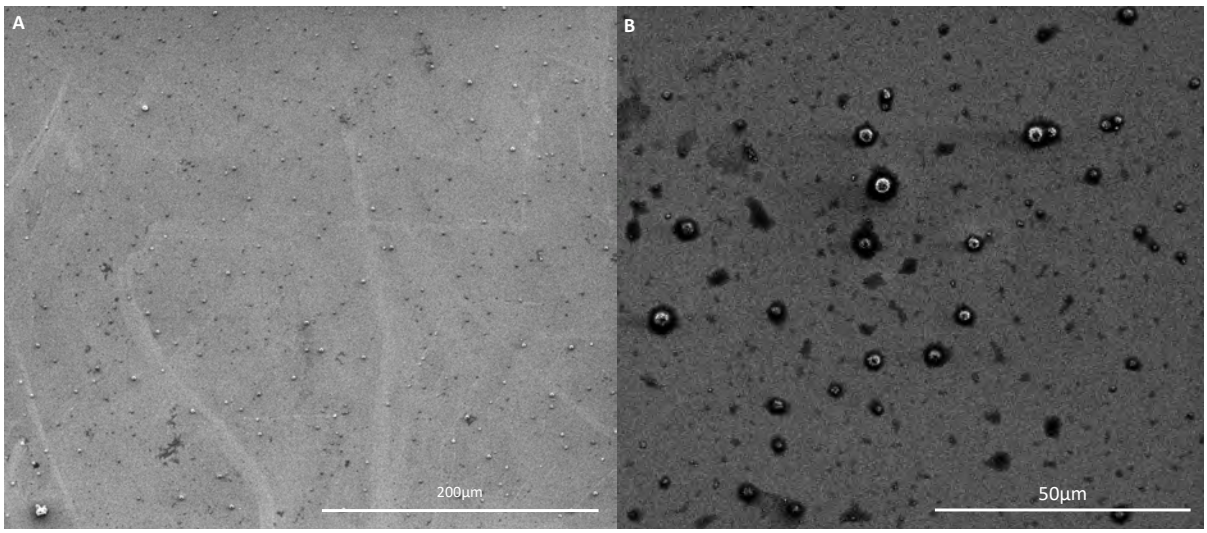

Figure S3. (A-B) SEM images at different magnifications showing the uniformity of C_{60} coated polystyrene beads on a silicon wafer, with the material generated using the optimized conditions: rotational speed (ω) 4k rpm under continuous-flow conditions, 0.1 mL min^{-1}, θ 45°, concentration of fullerene C_{60} and poly(styrene-co-divinylbenzene) beads in toluene 0.1 mg.mL^{-1}.

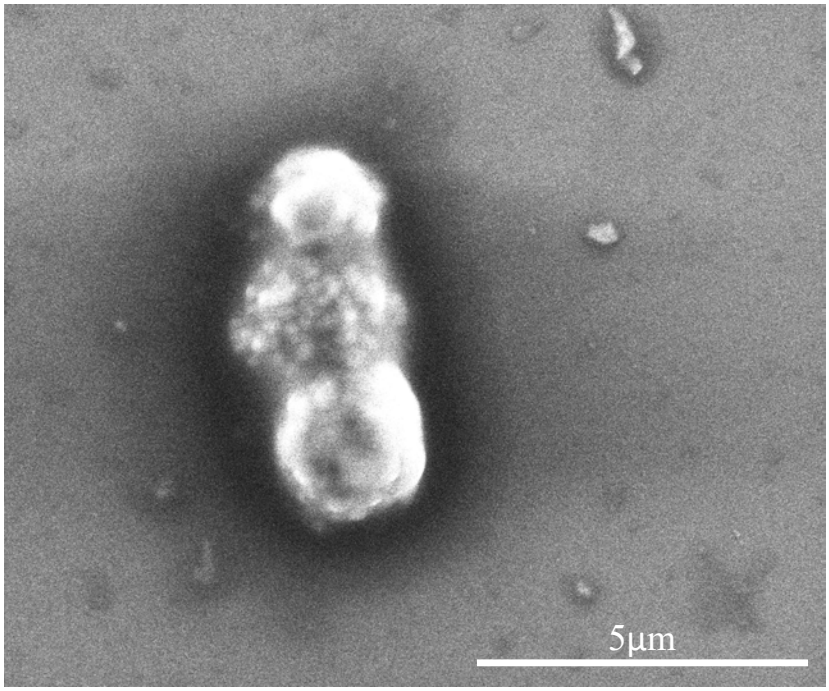

Figure S4. SEM image showing the partial coating of C_{60} over two polystyrene beads formed using the optimized conditions: rotational speed (ω) 4k rpm under continuous-flow conditions, 0.1 mL min^{-1}, θ = 45°, concentration of fullerene C_{60} and poly(styrene-co-divinylbenzene) beads 0.1 $mg.mL^{-1}$.

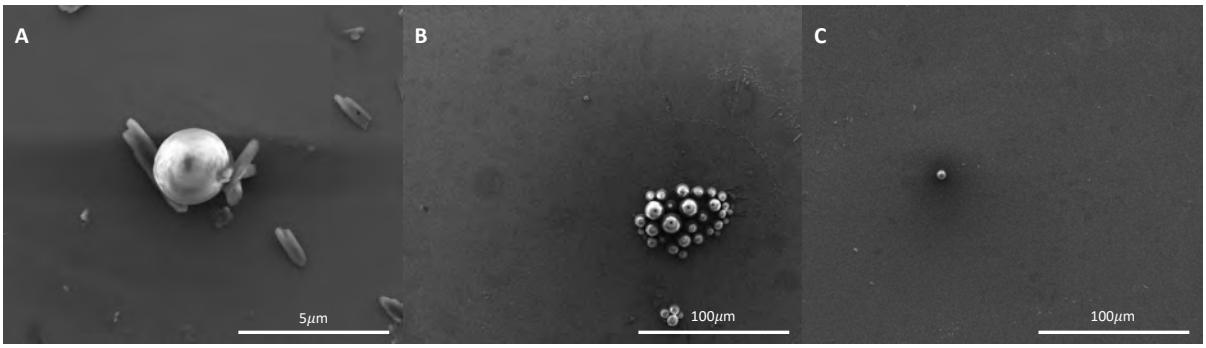

Figure S5. (A-C) SEM images of different magnification of polystyrene beads not coated with fullerene C_{60} after processing in the confined mode of operation of the VFD: rotational speed (ω) = 4k rpm, time = 30 minutes, θ = 45°, concentration of C_{60} and poly(styrene-co-divinylbenzene) both 0.1 $mg.mL^{-1}$ in toluene, after centrifugation at 1.751 RCF for 30 minutes, and the solid pellet collected on a filter paper.

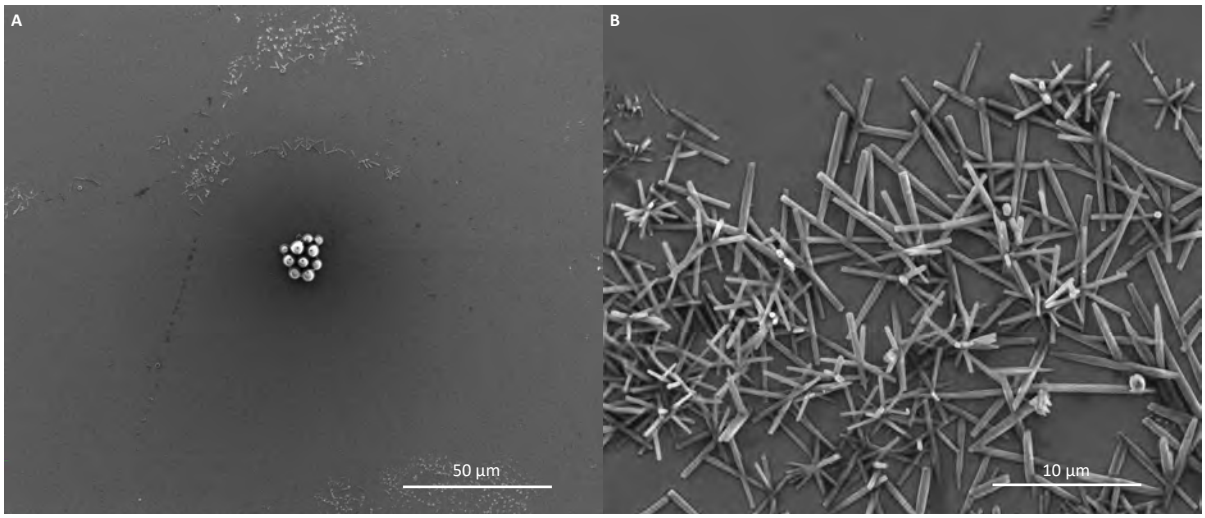

Figure S6. (A) SEM image of polystyrene beads devoid of coating with fullerene C_{60} after processing in confined mode of operation: rotational speed (ω) 7.5k rpm under confined mode conditions, time = 30 minutes, θ = 45°, concentration of fullerene C_{60} and poly(styrene-co-divinylbenzene) 0.1 mg.mL^{-1} in toluene. (B) SEM image of crystallised fullerene C_{60} after processing in the confined mode: rotational speed (ω) 7.5k rpm, time = 30 minutes, θ = 45°, concentration of fullerene C_{60} and poly(styrene-co-divinylbenzene) 0.1 mg.mL^{-1} in toluene.

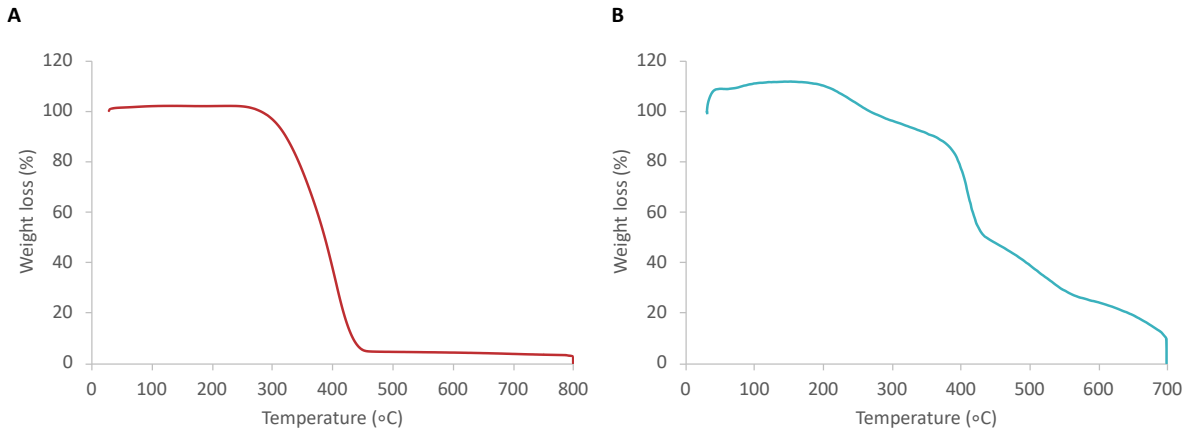

Figure S7. Thermal gravitational analysis (TGA) of (A) the as received polystyrene beads and (B) C_{60} coated polystyrene beads formed in the VFD at ω 4k rpm under continuous-flow conditions, flow rate (ν) 0.1 mL min^{-1}, θ 45°, concentration of fullerene C_{60} and poly(styrene-co-divinylbenzene) in toluene 0.1 mg mL^{-1}, operating under a nitrogen atmosphere with a temperature range 30 to 800 ºC (30 to 700 ºC for coated beads) at 10 ºC/min (approximate weight of samples was 2 to 4 mg).

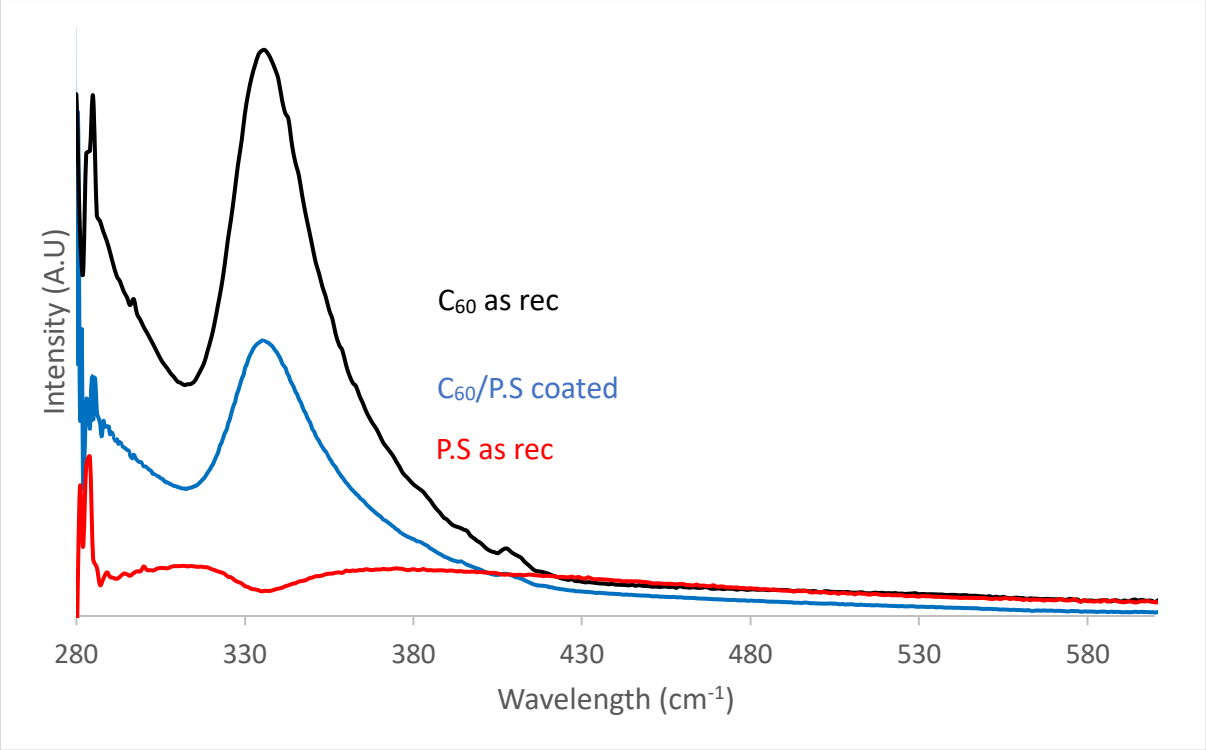

Figure S8. UV-Vis spectra of the as received C_{60} (black) (concentration 0.1 mg.mL^{-1}), coated polystyrene beads with C_{60} (blue) formed in the VFD at a rotational speed (ω) 4k rpm under continuous-flow conditions, 0.1 mL min^{-1}, θ = 45°, concentration of fullerene C_{60} and poly(styrene-co-divinylbenzene) beads in toluene 0.1 mg mL^{-1} and poly(styrene-co-divinylbenzene) as received (red) (concentration of 0.1mg.mL^{-1}).

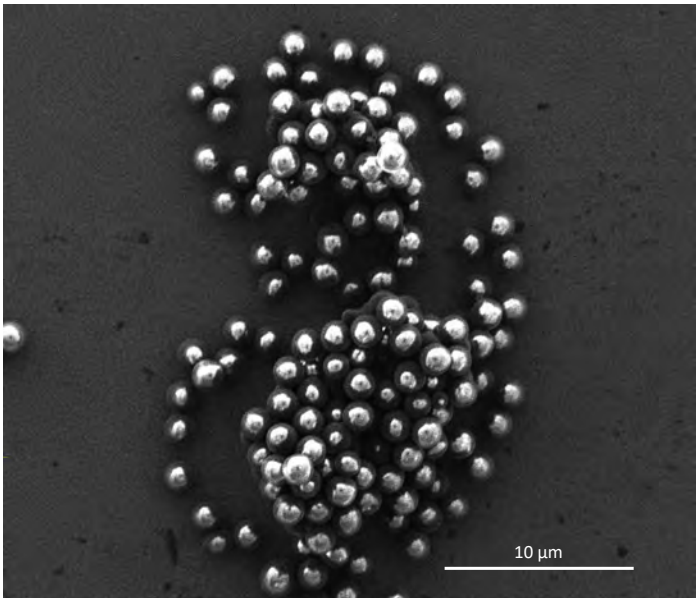

Figure S9. SEM image of a polystyrene bead (Avg. particle size ca. 14-20 µm) as received after dispersing in toluene, then drop casted on a silicon wafer.

Table 1: Summary of the different conditions explored in optimising the processing in the VFD.

C60 concentration (mg/mL)	Polystryene concentration (mg/mL)	Polystyrene size (um)	Rotational speed (krpm)	Tube diameter (O.D)	Tilt angle (θ)	VFD mode	Flow rate (mL/min)	Time (mins)	Yield (%)
0.1	0.1	6	4	20	45	confined	N/A	30	<10
0.1	0.1	6	7.5	20	45	confined	N/A	30	0
0.5	0.1	6	4	20	45	confined	N/A	30	<10 a
1	0.1	6	4	20	45	confined	N/A	31	<10 b
0.1	0.1	6	4	20	45	continous flow	0.1	N/A	>95
0.1	0.1	6	4	20	40	continous flow	0.1	N/A	>95
0.1	0.1	6	4	20	50	continous flow	0.1	N/A	>95
0.1	0.1	17	4	20	45	continous flow	0.1	N/A	0
0.1	0.1	6	4	10	45	continous flow	0.1	N/A	0

[a] 0.5 mg/mL concertation of C_{60} had minimal effect on the yield of coated beads, but there was the presence of spicules devoid of the beads.

[b] 1 mg/mL concertation of C_{60} had minimal effect on the yield of coated beads, but there was the presence of spicules devoid of the beads.

References

T. M. D. Alharbi, M. Jellicoe, X. Luo, K. Vimalanathan, I. K. Alsulami, B. AL Harbi, A. Igder, X. Chen, K. A. Stubbs, J. M. Chalker, W. Zhang, R. A. Boulos, D. B. Jones, J. S. Quinton, and C. L. Raston.
https://doi.org/10.26434/chemrxiv.13141352.v1