

## **Supporting Information**

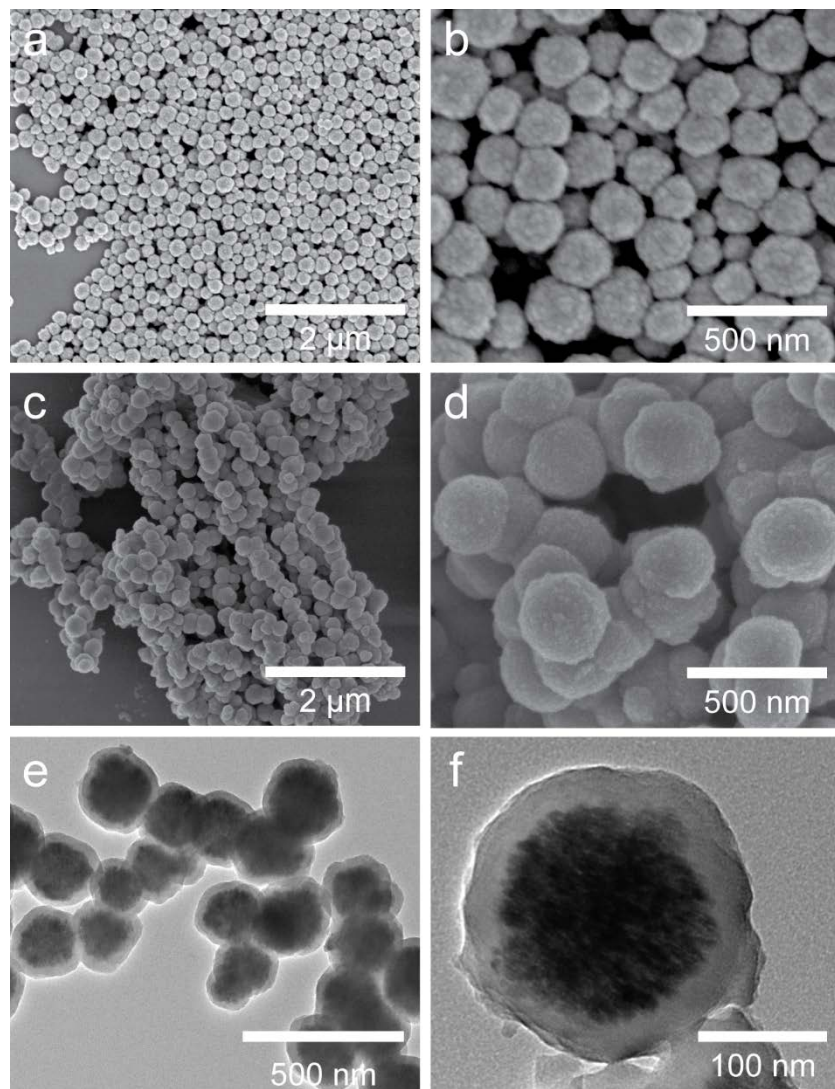
# **Facile Synthesis of Magnetic Mesoporous Hollow Carbon Microspheres for Rapid Capture of Low-concentration Peptides**

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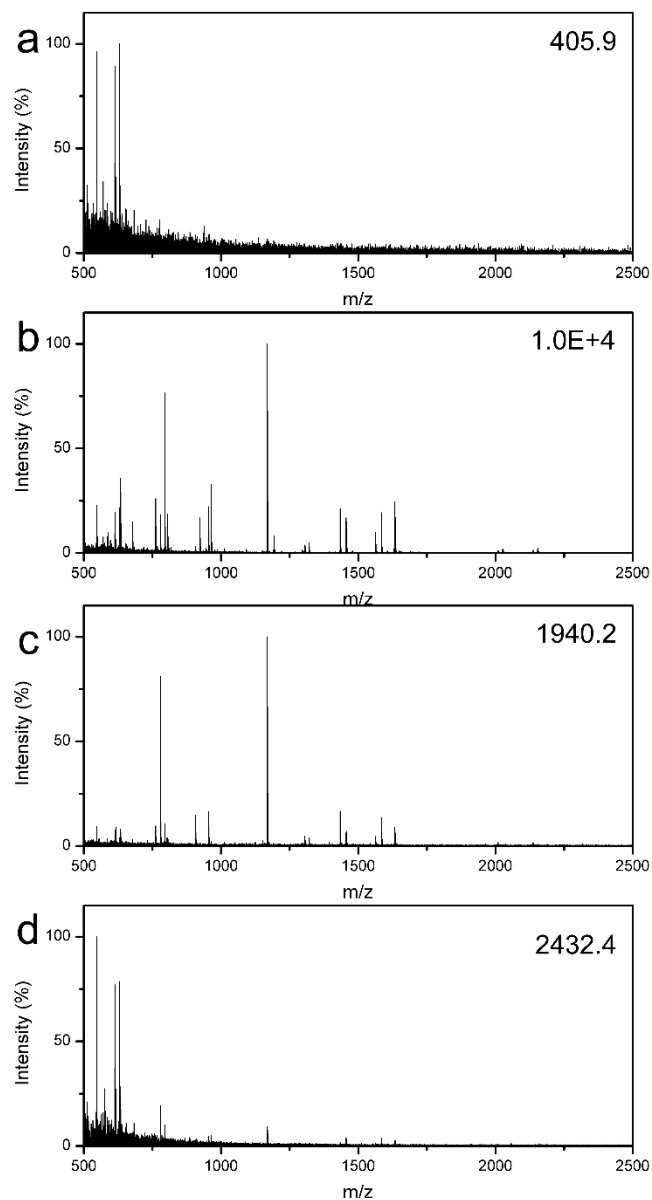
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**Figures:**



**Fig. S1** SEM (a-d) and TEM (e and f) images of prepared  $\text{Fe}_3\text{O}_4$  (a and b) and  $\text{Fe}_3\text{O}_4$ @PDA microspheres (c-f)



**Fig. S2** MALDI-TOF mass spectra of the diluted Cyt-C digest (2.5 nM): direct detection (a) and after enrichment with the MPC affinity microspheres (b), the ZipTipC18 pipette tip (c), and the magnetic mesoporous silica microspheres (d), respectively. The number in the top right corner is the highest peak intensity.

**Tables:****Table S1** The peptides captured from BSA digest by different affinity materials and identified by MALDI-TOF MS. M@mSiO<sub>2</sub> stands for magnetic mesoporous silica microspheres.

| Start-End | MW       | Sequence       | MHM | Zip-tip C18 | M@mSiO <sub>2</sub> |
|-----------|----------|----------------|-----|-------------|---------------------|
| 20-24     | 633.3711 | R.GVFRR.D      |     |             | ✓                   |
| 24-28     | 655.3402 | R.RDTHK.S      |     |             | ✓                   |
| 29-34     | 711.3664 | K.SEIAHR.F     | ✓   |             |                     |
| 35-44     | 1248.614 | R.FKDLGEEHFK.G | ✓   | ✓           | ✓                   |
| 37-44     | 973.4505 | K.DLGEEHFK.G   | ✓   |             |                     |
| 66-75     | 1162.623 | K.LVNELTEFAK.T | ✓   | ✓           | ✓                   |
| 101-105   | 544.3333 | K.VASLR.E      | ✓   | ✓           |                     |
| 118-122   | 657.3082 | K.QEPER.N      |     | ✓           |                     |
| 156-160   | 664.3697 | K.KFWGK.Y      | ✓   | ✓           | ✓                   |
| 157-160   | 536.2747 | K.FWGK.Y       | ✓   | ✓           |                     |
| 161-167   | 926.4861 | K.YLYEIAR.R    | ✓   | ✓           | ✓                   |
| 198-204   | 700.3942 | K.GACLLPK.I    | ✓   |             |                     |
| 219-222   | 572.3221 | R.QRLR.C       | ✓   | ✓           |                     |
| 223-228   | 648.3265 | R.CASIQK.F     | ✓   |             | ✓                   |
| 229-232   | 507.2441 | K.FGER.A       | ✓   | ✓           |                     |
| 236-241   | 688.3656 | K.AWSVAR.L     | ✓   | ✓           |                     |
| 242-248   | 846.4963 | R.LSQKFPK.A    | ✓   |             | ✓                   |
| 249-256   | 921.4807 | K.AEFVEVTK.L   | ✓   |             |                     |
| 257-263   | 788.4644 | K.LVTDLTK.V    | ✓   |             |                     |
| 281-285   | 516.2907 | R.ADLAK.Y      | ✓   | ✓           |                     |
| 341-346   | 751.35   | K.NYQEAK.D     | ✓   |             |                     |

|                       |          |                     |    |    |    |
|-----------------------|----------|---------------------|----|----|----|
| 347-359               | 1566.735 | K.DAFLGSFLYEYSR.R   | ✓  |    | ✓  |
| 360-371               | 1438.805 | R.RHPEYAVSVLLR.L    | ✓  | ✓  | ✓  |
| 361-371               | 1282.703 | R.HPEYAVSVLLR.L     | ✓  |    | ✓  |
| 402-412               | 1304.709 | K.HLVDEPQNLIK.Q     | ✓  | ✓  | ✓  |
| 421-433               | 1478.788 | K.LGEYGFQNALIVR.Y   | ✓  | ✓  | ✓  |
| 434-437               | 566.3176 | R.YTRK.V            | ✓  |    |    |
| 437-451               | 1638.931 | R.KVPQVSTPTLVEVSR.S | ✓  |    | ✓  |
| 438-451               | 1510.836 | K.VPQVSTPTLVEVSR.S  | ✓  |    | ✓  |
| 490-495               | 659.349  | K.TPVSEK.V          | ✓  | ✓  |    |
| 499-507               | 1023.448 | K.CCTESLVNR.R       | ✓  |    |    |
| 524-528               | 608.2806 | K.AFDEK.L           | ✓  |    | ✓  |
| 548-557               | 1141.707 | K.KQTALVELLK.H      | ✓  |    | ✓  |
| 549-557               | 1013.612 | K.QTALVELLK.H       | ✓  |    | ✓  |
| 558-561               | 508.3121 | K.HKPK.A            | ✓  |    |    |
| 562-568               | 817.4181 | K.ATEEQLK.T         | ✓  |    |    |
| 598-607               | 1001.576 | K.LVVSTQTALA.-      | ✓  |    |    |
| Matched peptides      |          |                     | 34 | 16 | 19 |
| Sequence coverage (%) |          |                     | 37 | 20 | 22 |

**Table S2** The peptides captured from Cyt-c digest by different affinity materials and identified by MALDI-TOF MS

| Start-End | MW       | Peptide Sequence | MHM | Zip-tip | M@mSiO <sub>2</sub> |
|-----------|----------|------------------|-----|---------|---------------------|
| 1-6       | 677.3054 | -.MGDVEK.G       | ✓   |         |                     |
| 2-6       | 546.264  | M.GDVEK.G        | ✓   | ✓       | ✓                   |
| 9-14      | 761.4800 | K.KIFVQK.C       | ✓   |         | ✓                   |
| 10-14     | 633.3850 | K.IFVQK.C        | ✓   | ✓       | ✓                   |

|                       |           |                        |    |    |    |
|-----------------------|-----------|------------------------|----|----|----|
| 10-23                 | 1632.8116 | K.IFVQKCAQCHTVEK.G     | ✓  | ✓  | ✓  |
| 24-28                 | 525.3023  | K.GGKHK.T              |    | ✓  |    |
| 27-39                 | 1432.768  | K.HKTGPNLHGLFGR.K      | ✓  |    | ✓  |
| 29-39                 | 1167.6149 | K.TGPNLHGLFGR.K        | ✓  | ✓  | ✓  |
| 40-54                 | 1583.7580 | R.KTGQAPGFSYTDANK.N    | ✓  | ✓  | ✓  |
| 41-54                 | 1455.6630 | K.TGQAPGFSYTDANK.N     | ✓  | ✓  | ✓  |
| 57-73                 | 2008.9452 | K.GITWGEETLMEYLENPK.K  | ✓  |    | ✓  |
| 57-74                 | 2137.0401 | K.GITWGEETLMEYLENPKK.Y | ✓  |    | ✓  |
| 74-80                 | 805.4698  | K.KYIPGTK.M            | ✓  |    | ✓  |
| 81-87                 | 778.4411  | K.MIFAGIK.K            | ✓  | ✓  | ✓  |
| 81-88                 | 906.5361  | K.MIFAGIKK.K           | ✓  |    |    |
| 90-100                | 1305.6928 | K.GEREDLIAYLK.K        | ✓  |    | ✓  |
| 93-100                | 963.5277  | R.EDLIAYLK.K           | ✓  |    |    |
| 93-101                | 1091.622  | R.EDLIAYLKK.A          | ✓  |    |    |
| 101-105               | 561.2758  | K.KATNE.-              | ✓  | ✓  |    |
| Matched peptides      |           |                        | 19 | 9  | 14 |
| Sequence coverage (%) |           |                        | 92 | 59 | 86 |

**Table S3** The peptides captured from human urine

| m/z   | S/N  | m/z    | S/N  |
|-------|------|--------|------|
| 757.4 | 33.2 | 2212.7 | 65.6 |
| 982.6 | 39.4 | 2216.7 | 19.9 |

|        |       |        |        |
|--------|-------|--------|--------|
| 1148.5 | 10.7  | 2228.7 | 16.2   |
| 1188.7 | 51.7  | 2436.8 | 102.2  |
| 1204.7 | 104.8 | 2674.9 | 12.3   |
| 1218.6 | 11.9  | 2787.9 | 513.9  |
| 1251.6 | 11.6  | 2790.9 | 270.43 |
| 1406.5 | 11.4  | 2805.9 | 17.9   |
| 1912.1 | 125.1 | 2809.9 | 56.21  |
| 2079.7 | 10.2  | 2814.9 | 19.68  |
| 2192.8 | 278.5 | 2825.9 | 21.0   |