

## Supplementary Materials

### Formation of Phases and Porous System in the Product of Hydrothermal Treatment of $\chi$ -Al<sub>2</sub>O<sub>3</sub>

**Table S1.** TG/DSC/MS analyses results of starting  $\chi$ -Al<sub>2</sub>O<sub>3</sub> and the products of its hydrothermal treatment.

| Treatment Conditions                   |                                         | Endothermic Effects                                               |                                                             |                                |                                                              |                                |                                                               |                                | Mass Loss in The Temperature Range According to The TG Data, % |         |         |         |         |
|----------------------------------------|-----------------------------------------|-------------------------------------------------------------------|-------------------------------------------------------------|--------------------------------|--------------------------------------------------------------|--------------------------------|---------------------------------------------------------------|--------------------------------|----------------------------------------------------------------|---------|---------|---------|---------|
| <i>T</i> (pH), °C                      | $\tau$ , min                            | I Effect<br>(H <sub>2</sub> O* <sup>Physically</sup><br>Adsorbed) | II Effect<br>(H <sub>2</sub> O** <sup>Gb</sup> Dehydration) |                                | III Effect<br>(H <sub>2</sub> O*** <sup>Encapsulated</sup> ) |                                | IV Effect<br>(H <sub>2</sub> O**** <sup>Bm</sup> Dehydration) |                                | 30–200                                                         | 200–375 | 375–440 | 440–600 | 30–1000 |
|                                        |                                         | <i>T</i> <sub>peak</sub> , °C                                     | <i>T</i> <sub>peak</sub> , °C                               | $\Delta T$ , °C<br>(Start–End) | <i>T</i> <sub>peak</sub> , °C                                | $\Delta T$ , °C<br>(Start–End) | <i>T</i> <sub>peak</sub> , °C                                 | $\Delta T$ , °C<br>(Start–End) |                                                                |         |         |         |         |
| $\chi$ -Al <sub>2</sub> O <sub>3</sub> |                                         | 108                                                               | –                                                           | –                              | –                                                            | –                              | –                                                             | –                              | 7.1                                                            | 2.0     | 0.6     | 0.4     | 10.2    |
| 150(9.2)                               | 20 <sup>rise in <i>T</i> (119 °C)</sup> | 95                                                                | 270                                                         | 242–289                        | –                                                            | –                              | –                                                             | –                              | 3.7                                                            | 2.1     | 1.0     | 0.8     | 8.2     |
|                                        | 40 <sup>rise in <i>T</i> (143 °C)</sup> | 102                                                               | 281                                                         | 246–296                        | –                                                            | –                              | –                                                             | –                              | 4.0                                                            | 2.4     | 0.9     | 0.8     | 8.6     |
|                                        | 0 <sup>attaining of <i>T</i></sup>      | 95                                                                | 289                                                         | 250–304                        | –                                                            | –                              | –                                                             | –                              | 3.8                                                            | 4.7     | 0.7     | 0.5     | 11.3    |
|                                        | 30                                      | 99                                                                | 264                                                         | 249–357                        | –                                                            | –                              | 496                                                           | 454–519                        | 4.7                                                            | 1.3     | –       | 2.3     | 12.0    |
|                                        | 60                                      | 103                                                               | 267                                                         | 251–370                        | –                                                            | –                              | 498                                                           | 457–520                        | 4.4                                                            | 2.3     | –       | 2.4     | 11.5    |
|                                        | 90                                      | 99                                                                | 266                                                         | 250–355                        | –                                                            | –                              | 500                                                           | 466–522                        | 3.7                                                            | 2.2     | –       | 3.3     | 11.9    |
|                                        | 120                                     | 101                                                               | 266                                                         | 252–362                        | –                                                            | –                              | 503                                                           | 470–525                        | 3.1                                                            | 2.0     | –       | 4.5     | 12.1    |
|                                        | 150                                     | 105                                                               | 268                                                         | 253–366                        | –                                                            | –                              | 506                                                           | 474–529                        | 3.5                                                            | 1.8     | –       | 4.8     | 12.4    |
|                                        | 180                                     | 103                                                               | 265                                                         | 238–359                        | –                                                            | –                              | 506                                                           | 475–531                        | 2.9                                                            | 1.7     | –       | 5.3     | 12.6    |
| 180(9.2)                               | 20 <sup>rise in <i>T</i> (123 °C)</sup> | 93                                                                | 278                                                         | 244–296                        | –                                                            | –                              | –                                                             | –                              | 4.3                                                            | 3.2     | –       | 1.0     | 9.8     |
|                                        | 40 <sup>rise in <i>T</i> (161 °C)</sup> | 96                                                                | 292                                                         | 252–307                        | –                                                            | –                              | 502                                                           | 473–565                        | 3.5                                                            | 5.6     | –       | 1.9     | 12.4    |
|                                        | 0 <sup>attaining <i>T</i></sup>         | 102                                                               | 267                                                         | 251–284                        | –                                                            | –                              | 506                                                           | 475–529                        | 3.8                                                            | 1.9     | 0.7     | 3.7     | 12.4    |
|                                        | 15                                      | 97                                                                | 261                                                         | 246–278                        | –                                                            | –                              | 507                                                           | 476–530                        | 3.9                                                            | 1.8     | 0.7     | 4.2     | 12.7    |
|                                        | 30                                      | 94                                                                | 259                                                         | 244–275                        | –                                                            | –                              | 508                                                           | 478–570                        | 3.7                                                            | 2.0     | 0.8     | 4.9     | 13.5    |
|                                        | 60                                      | 93                                                                | 256                                                         | 244–273                        | 400                                                          | 386–422                        | 512                                                           | 482–535                        | 2.7                                                            | 2.2     | 0.8     | 5.8     | 13.8    |
|                                        | 90                                      | 89                                                                | 254 <sup>P</sup>                                            | –                              | 401                                                          | 385–425                        | 518                                                           | 485–590                        | 2.2                                                            | 1.7     | 1.2     | 7.1     | 14.9    |
|                                        | 120                                     | 84                                                                | 256 <sup>P</sup>                                            | –                              | 400                                                          | 385–429                        | 528                                                           | 489–600                        | 1.3                                                            | 1.2     | 1.2     | 9.6     | 15.6    |
|                                        | 150                                     | 76                                                                | 265 <sup>P</sup>                                            | –                              | 400                                                          | 385–422                        | 536                                                           | 492–563                        | 0.6                                                            | 1.1     | 1.1     | 11.1    | 16.2    |
|                                        | 180                                     | 74                                                                | 267 <sup>P</sup>                                            | –                              | 402                                                          | 386–426                        | 540                                                           | 496–600                        | 0.2                                                            | 1.0     | 1.0     | 12.5    | 16.5    |

Table S1. *Cont.*

| Treatment Conditions |                                               | Endothermic Effects                                   |                                                 |                                |                                                 |                                |                                                   |                                | Mass Loss in The Temperature Range According to The TG Data, % |         |         |         |         |
|----------------------|-----------------------------------------------|-------------------------------------------------------|-------------------------------------------------|--------------------------------|-------------------------------------------------|--------------------------------|---------------------------------------------------|--------------------------------|----------------------------------------------------------------|---------|---------|---------|---------|
| $T(\text{pH})$ , °C  | $\tau$ , min                                  | I Effect<br>(H <sub>2</sub> O*Physically<br>Adsorbed) | II Effect<br>(H <sub>2</sub> O**Gb Dehydration) |                                | III Effect<br>(H <sub>2</sub> O***Encapsulated) |                                | IV Effect<br>(H <sub>2</sub> O****Bm Dehydration) |                                | 30–200                                                         | 200–375 | 375–440 | 440–600 | 30–1000 |
|                      |                                               | $T_{\text{peak}}$ , °C                                | $T_{\text{peak}}$ , °C                          | $\Delta T$ , °C<br>(Start–End) | $T_{\text{Peak}}$ , °C                          | $\Delta T$ , °C<br>(Start–End) | $T_{\text{Peak}}$ , °C                            | $\Delta T$ , °C<br>(Start–End) |                                                                |         |         |         |         |
| 190(9.2)             | 20 <sup>rise in <math>T</math> (127 °C)</sup> | 93                                                    | 284                                             | 247–299                        | –                                               | –                              | –                                                 | –                              | 4.2                                                            | 3.6     | –       | 1.0     | 10.4    |
|                      | 40 <sup>rise in <math>T</math> (173 °C)</sup> | 93                                                    | 283                                             | 252–296                        | –                                               | –                              | 504                                               | 475–529                        | 3.3                                                            | 2.4     | –       | 1.4     | 8.7     |
|                      | 0 <sup>attaining <math>T</math></sup>         | 98                                                    | 262                                             | 246–367                        | 400                                             | 386–430                        | 510                                               | 479–539                        | 3.7                                                            | 1.9     | 0.7     | 3.6     | 13.0    |
|                      | 15                                            | 96                                                    | 259                                             | 179–366                        | 400                                             | 388–428                        | 514                                               | 482–550                        | 2.9                                                            | 1.9     | 1.1     | 5.3     | 13.7    |
|                      | 30                                            | 89                                                    | 254                                             | 184–268                        | 401                                             | 387–425                        | 511                                               | 389–534                        | 2.9                                                            | 2.1     | 1.0     | 5.4     | 13.9    |
|                      | 60                                            | 89                                                    | 256 <sup>P</sup>                                | –                              | 402                                             | 388–426                        | 522                                               | 486–548                        | 1.8                                                            | 1.2     | 1.5     | 7.6     | 14.9    |
|                      | 90                                            | 79                                                    | 249 <sup>P</sup>                                | –                              | 403                                             | 391–422                        | 538                                               | 492–566                        | 0.7                                                            | 1.2     | 1.2     | 10.9    | 15.9    |
|                      | 120                                           | 70                                                    | 236 <sup>P</sup>                                | –                              | 406                                             | 388–430                        | 541                                               | 496–569                        | 0.2                                                            | 0.5     | 1.1     | 12.6    | 16.7    |
|                      | 150                                           | 69                                                    | 249 <sup>P</sup>                                | –                              | 407                                             | 389–437                        | 545                                               | 498–573                        | 0.1                                                            | 0.4     | 1.1     | 13.0    | 16.9    |
|                      | 180                                           | 69                                                    | 257 <sup>P</sup>                                | –                              | 403                                             | 387–437                        | 543                                               | 497–596                        | 0.1                                                            | 0.3     | 1.1     | 13.9    | 16.9    |
| 200(9.2)             | 20 <sup>rise in <math>T</math> (137 °C)</sup> | 100                                                   | 284                                             | 249–303                        | –                                               | –                              | –                                                 | –                              | 3.5                                                            | 3.5     | –       | 1.1     | 9.4     |
|                      | 40 <sup>rise in <math>T</math> (182 °C)</sup> | 98                                                    | 284                                             | 251–299                        | –                                               | –                              | 507                                               | 476–535                        | 3.4                                                            | 3.2     | 0.6     | 1.3     | 9.8     |
|                      | 0 <sup>attaining <math>T</math></sup>         | 96                                                    | 260                                             | 182–276                        | 399                                             | 389–422                        | 514                                               | 482–546                        | 3.1                                                            | 2.2     | 1.2     | 5.8     | 13.7    |
|                      | 15                                            | 93                                                    | 257                                             | 240–363                        | 398                                             | 390–424                        | 516                                               | 484–539                        | 2.4                                                            | 1.9     | 1.5     | 6.9     | 14.0    |
|                      | 30                                            | 89                                                    | 255 <sup>P</sup>                                | –                              | 401                                             | 392–428                        | 524                                               | 488–550                        | 1.9                                                            | 1.5     | 1.3     | 8.9     | 14.9    |
|                      | 60                                            | 80                                                    | 253 <sup>P</sup>                                | –                              | 405                                             | 389–431                        | 537                                               | 491–562                        | 0.9                                                            | 0.8     | 1.2     | 12.0    | 15.8    |
|                      | 90                                            | 73                                                    | 250 <sup>P</sup>                                | –                              | 406                                             | 391–438                        | 545                                               | 498–573                        | 0.3                                                            | 0.5     | 1.1     | 13.2    | 16.2    |
|                      | 120                                           | 71                                                    | 244 <sup>P</sup>                                | –                              | 410                                             | 390–438                        | 544                                               | 498–571                        | 0.2                                                            | 0.4     | 1.1     | 13.7    | 16.6    |
|                      | 150                                           | 68                                                    | 240 <sup>P</sup>                                | –                              | 413                                             | 392–436                        | 544                                               | 496–571                        | 0.1                                                            | 0.4     | 1.0     | 14.0    | 16.7    |
|                      | 180                                           | 60                                                    | –                                               | –                              | 401                                             | 387–418                        | 539                                               | 497–559                        | 0.1                                                            | 0.4     | 0.8     | 14.5    | 16.7    |
|                      | 180****                                       | 60                                                    | –                                               | –                              | –                                               | –                              | 542                                               | 497–565                        | 0.1                                                            | 0.3     | –       | 4.5     | 16.2    |

Table S1. *Cont.*

| Treatment Conditions |                                  | Endothermic Effects                                             |                                                              |                                |                                                              |                                |                                                                |                                | Mass Loss in The Temperature Range According to The TG Data, % |         |         |         |         |
|----------------------|----------------------------------|-----------------------------------------------------------------|--------------------------------------------------------------|--------------------------------|--------------------------------------------------------------|--------------------------------|----------------------------------------------------------------|--------------------------------|----------------------------------------------------------------|---------|---------|---------|---------|
| T(pH), °C            | $\tau$ , min                     | I Effect<br>(H <sub>2</sub> O* <sup>Physically Adsorbed</sup> ) | II Effect<br>(H <sub>2</sub> O** <sup>Gb Dehydration</sup> ) |                                | III Effect<br>(H <sub>2</sub> O*** <sup>Encapsulated</sup> ) |                                | IV Effect<br>(H <sub>2</sub> O**** <sup>Bm Dehydration</sup> ) |                                | 30–200                                                         | 200–375 | 375–440 | 440–600 | 30–1000 |
|                      |                                  | $T_{peak}$ , °C                                                 | $T_{peak}$ , °C                                              | $\Delta T$ , °C<br>(Start–End) | $T_{peak}$ , °C                                              | $\Delta T$ , °C<br>(Start–End) | $T_{peak}$ , °C                                                | $\Delta T$ , °C<br>(Start–End) |                                                                |         |         |         |         |
|                      |                                  |                                                                 |                                                              |                                |                                                              |                                |                                                                |                                |                                                                |         |         |         |         |
| 190(4.0)             | 20 <sup>rise in T (129 °C)</sup> | 93                                                              | 277                                                          | 248–298                        | –                                                            | –                              | –                                                              | –                              | 3.2                                                            | 2.2     | 1.1     | 1.9     | 9.7     |
|                      | 40 <sup>rise in T (171 °C)</sup> | 95                                                              | 289                                                          | 258–301                        | –                                                            | –                              | –                                                              | –                              | 2.8                                                            | 2.6     | 1.0     | 2.1     | 10.6    |
|                      | 0 <sup>attaining T</sup>         | 98                                                              | 278                                                          | 179–354                        | –                                                            | –                              | 495                                                            | 445–520                        | 3.9                                                            | 2.6     | –       | 4.6     | 12.7    |
|                      | 15                               | 99                                                              | 274                                                          | 216–323                        | –                                                            | –                              | 485                                                            | 412–573                        | 3.8                                                            | 2.4     | –       | 4.9     | 12.2    |
|                      | 45                               | 99                                                              | 271                                                          | 185–313                        | –                                                            | –                              | 501                                                            | 411–532                        | 4.0                                                            | 2.5     | –       | 5.8     | 13.6    |
|                      | 60                               | 96                                                              | 272                                                          | 180–323                        | –                                                            | –                              | 503                                                            | 411–535                        | 3.9                                                            | 2.4     | –       | 6.2     | 13.6    |
|                      | 75                               | 97                                                              | 278                                                          | 186–315                        | –                                                            | –                              | 507                                                            | 414–535                        | 4.3                                                            | 2.5     | –       | 6.5     | 14.5    |
|                      | 90                               | 95                                                              | 280                                                          | 181–357                        | –                                                            | –                              | 507                                                            | 412–535                        | 3.6                                                            | 2.4     | –       | 6.9     | 14.1    |
|                      | 120                              | 96                                                              | 279                                                          | 179–292                        | –                                                            | –                              | 510                                                            | 411–536                        | 3.4                                                            | 2.3     | –       | 7.4     | 14.4    |
|                      | 150                              | 90                                                              | 278                                                          | 176–310                        | –                                                            | –                              | 508                                                            | 430–534                        | 3.8                                                            | 2.3     | –       | 7.2     | 14.7    |
| 190(6.0)             | 180                              | 92                                                              | 285                                                          | 172–326                        | –                                                            | –                              | 514                                                            | 417–540                        | 2.9                                                            | 2.0     | –       | 8.8     | 15.2    |
|                      | 0 <sup>attaining T</sup>         | 115                                                             | 267                                                          | 253–284                        | 385                                                          | 378–411                        | 509                                                            | 480–534                        | 2.8                                                            | 2.3     | 1.0     | 3.6     | 11.2    |
|                      | 15                               | 114                                                             | 266                                                          | 251–283                        | 382                                                          | 379–404                        | 512                                                            | 485–536                        | 2.5                                                            | 1.8     | 1.0     | 4.3     | 11.9    |
|                      | 30                               | 103                                                             | 257                                                          | 244–273                        | 385                                                          | 378–405                        | 513                                                            | 480–536                        | 2.7                                                            | 2.0     | 1.0     | 5.1     | 12.8    |
|                      | 60                               | 99                                                              | 255                                                          | 188–362                        | 387                                                          | 379–408                        | 517                                                            | 484–541                        | 2.0                                                            | 1.9     | 1.2     | 7.1     | 13.8    |
|                      | 90                               | 96                                                              | 264 <sup>P</sup>                                             | –                              | 390                                                          | 379–403                        | 524                                                            | 483–550                        | 1.7                                                            | 1.5     | 1.2     | 9.2     | 14.9    |
|                      | 120                              | 91                                                              | 262 <sup>P</sup>                                             | –                              | 392                                                          | 378–407                        | 528                                                            | 489–559                        | 0.9                                                            | 1.0     | 1.2     | 11.2    | 15.7    |
|                      | 150                              | 79                                                              | 253 <sup>P</sup>                                             | –                              | 393                                                          | 377–403                        | 539                                                            | 493–567                        | 0.4                                                            | 0.8     | 1.0     | 12.5    | 16.1    |
| 190(8.0)             | 180                              | 73                                                              | 273 <sup>P</sup>                                             | –                              | 394                                                          | 376–405                        | 540                                                            | 495–568                        | 0.1                                                            | 0.5     | 1.0     | 13.4    | 16.3    |
|                      | 0 <sup>attaining T</sup>         | 100                                                             | 264                                                          | 248–280                        | 398                                                          | 384–410                        | 512                                                            | 479–548                        | 3.8                                                            | 2.3     | 1.0     | 4.7     | 13.1    |
|                      | 15                               | 101                                                             | 264                                                          | 247–280                        | 393                                                          | 385–402                        | 514                                                            | 482–538                        | 3.2                                                            | 2.0     | 1.5     | 5.1     | 13.2    |
|                      | 30                               | 95                                                              | 259                                                          | 245–274                        | 397                                                          | 386–405                        | 551                                                            | 483–539                        | 2.9                                                            | 2.2     | 1.0     | 7.5     | 14.0    |
|                      | 60                               | 92                                                              | 260 <sup>P</sup>                                             | –                              | 392                                                          | 387–406                        | 526                                                            | 486–551                        | 1.7                                                            | 1.5     | 1.5     | 9.1     | 15.0    |
|                      | 90                               | 83                                                              | 270 <sup>P</sup>                                             | –                              | 399                                                          | 389–415                        | 535                                                            | 490–562                        | 0.9                                                            | 0.9     | 1.3     | 11.5    | 15.8    |
|                      | 120                              | 73                                                              | 297 <sup>P</sup>                                             | –                              | 402                                                          | 384–416                        | 542                                                            | 496–570                        | 0.2                                                            | 0.5     | 1.1     | 13.5    | 16.4    |
|                      | 150                              | 69                                                              | 252 <sup>P</sup>                                             | –                              | 402                                                          | 387–417                        | 545                                                            | 497–573                        | 0.1                                                            | 0.3     | 1.1     | 14.0    | 16.9    |
| 190(10.0)            | 150****                          | 60                                                              | –                                                            | –                              | –                                                            | –                              | 542                                                            | 497–565                        | 0.1                                                            | 0.3     | –       | 14.0    | 16.2    |

H<sub>2</sub>O\*<sup>physically adsorbed</sup>: release of physically adsorbed water; H<sub>2</sub>O\*\*<sup>Gb dehydration</sup>: water release during dehydration of gibbsite; H<sub>2</sub>O\*\*\*<sup>encapsulated</sup>: release of encapsulated water;

H<sub>2</sub>O\*\*\*\*<sup>Bm dehydration</sup>: water release during boehmite dehydration; \*\*\*\*\*: after additional heating up to 400 °C (10 °C / min);  $T_{peak}$ : water release from open mesopores.

**Table S2.** Mesoporous system parameters of  $\chi$ -Al<sub>2</sub>O<sub>3</sub> and the products of its hydrothermal treatment.

| Treatment Conditions                   |           | S <sub>BET</sub> ,<br>m <sup>2</sup> /g | V <sub>BET</sub> ,<br>cm <sup>3</sup> /g | V <sub>BJH</sub> ,<br>cm <sup>3</sup> /g | Distribution of V <sub>BJH</sub> on D <sub>p</sub> (%) |            |           | D <sub>max</sub> /((dV/dD)×10 <sup>2</sup> ) in Area of Pore Diameters, nm |                    |                      | Type of Hysteresis Loop |
|----------------------------------------|-----------|-----------------------------------------|------------------------------------------|------------------------------------------|--------------------------------------------------------|------------|-----------|----------------------------------------------------------------------------|--------------------|----------------------|-------------------------|
| T(pH),<br>°C                           | τ,<br>min |                                         |                                          |                                          | <3 nm                                                  | 3–5 nm     | >5 nm     | 2.0–3.0                                                                    | 3.1–5.0            | >5.0                 |                         |
| $\chi$ -Al <sub>2</sub> O <sub>3</sub> |           | 253                                     | 0.41                                     | 0.43                                     | 0.10(24)                                               | 0.15(35)   | 0.18(41)  | 2.5/55                                                                     | 3.4/1.86           | –/–                  | II–H3*                  |
| 150(9.2)                               | 120       | 226                                     | 0.36                                     | 0.38                                     | 0.05(12)                                               | 0.2254(60) | 0.11(28)  | 2.5/3.99                                                                   | 3.5/29.56          | –/–                  | II–H3*                  |
|                                        | 180       | 209                                     | 0.39                                     | 0.41                                     | 0.03(6)                                                | 0.26(63)   | 0.13(31)  | 2.5/2.73                                                                   | 4.0/20.32          | –/–                  | II–H3*                  |
| 180(9.2)                               | 30        | 226                                     | 0.42                                     | 0.44                                     | 0.02(6)                                                | 0.29(66)   | 0.13(28)  | 2.4/4.24                                                                   | 4.0/30.11          | –/–                  | II–H3*                  |
|                                        | 60        | 158                                     | 0.36                                     | 0.38                                     | 0.01(2)                                                | 0.26(69)   | 0.11(29)  | 2.5/0.93                                                                   | 4.2/6.60           | 5.9/4.79             | II–H3*                  |
|                                        | 90        | 109                                     | 0.32                                     | 0.34                                     | 0(0)                                                   | 0.18(53)   | 0.16(47)  | 2.7/0.22                                                                   | 3.7/1.57; 4.4/1.80 | 6.3/2.86; 7.4/2.83   | II–H3*                  |
|                                        | 120       | 61                                      | 0.21                                     | 0.21                                     | 0(0)                                                   | 0.08(37)   | 0.13(63)  | 2.5/0.12                                                                   | 3.7/0.92           | 10.3/1.40            | II–H3                   |
|                                        | 150       | 25                                      | 0.09                                     | 0.09                                     | 0(0)                                                   | 0.03(27)   | 0.06(73)  | 2.6/1.63                                                                   | 3.7/0.62           | 10.0/0.30            | II–H3                   |
|                                        | 180       | 15                                      | 0.05                                     | 0.06                                     | 0(0)                                                   | 0.01(19)   | 0.05(81)  | 2.6/0.23                                                                   | 3.7/0.32           | 10.0/0.08            | II–H3                   |
| 190(9.2)                               | 30        | 159                                     | 0.36                                     | 0.37                                     | 0.01(2)                                                | 0.26(70)   | 0.11(28)  | 2.6/0.96                                                                   | 4.5/6.76           | 5.5/6.58             | II–H3*                  |
|                                        | 60        | 114                                     | 0.33                                     | 0.34                                     | 0(0)                                                   | 0.17(50)   | 0.17(50)  | 2.6/0.63                                                                   | 3.7/1.71           | 6.3/2.86; 13.3/1.37  | II–H3                   |
|                                        | 90        | 31                                      | 0.11                                     | 0.11                                     | 0(0)                                                   | 0.03(32)   | 0.08(68)  | 2.9/0.24                                                                   | 3.7/0.64; 4.7/0.39 | 10.2/0.55; 11.6/0.55 | II–H3                   |
|                                        | 120       | 10                                      | 0.03                                     | 0.03                                     | 0(0)                                                   | 0.01(32)   | 0.02(68)  | 2.0/0.24                                                                   | 3.4/0.28; 4.6/0.14 | –/–                  | II–H3                   |
|                                        | 180       | 6                                       | 0.03                                     | 0.03                                     | 0(0)                                                   | 0(0)       | 0.03(100) | 2.0/0.11; 2.5/0.16                                                         | 3.5/0.15           | –/–                  | II–H3                   |
| 200(9.2)                               | 0         | 169                                     | 0.34                                     | 0.35                                     | 0.01(0)                                                | 0.25(73)   | 0.10(27)  | 2.6/14.06                                                                  | 4.8/11.26          | –/–                  | II–H3*                  |
|                                        | 15        | 135                                     | 0.35                                     | 0.36                                     | 0.01(2)                                                | 0.24(67)   | 0.11(31)  | 2.9/0.99                                                                   | 3.9/2.94; 4.8/4.65 | 5.4/4.22             | II–H3*                  |
|                                        | 30        | 95                                      | 0.29                                     | 0.30                                     | 0(0)                                                   | 0.14(50)   | 0.16(53)  | –/–                                                                        | 3.2/0.46; 3.7/1.11 | 6.8/2.29; 9.1/0.22   | II–H3                   |
|                                        | 60        | 39                                      | 0.14                                     | 0.14                                     | 0(0)                                                   | 0.04(30)   | 0.10(70)  | 2.9/0.19                                                                   | 3.7/0.55           | 11.6/0.91            | II–H3                   |
|                                        | 90        | 17                                      | 0.05                                     | 0.05                                     | 0(0)                                                   | 0.01(28)   | 0.04(72)  | 2.1/0.25                                                                   | 3.9/0.39           | 10.0/0.19; 13.3/0.18 | II–H3                   |
|                                        | 120       | 9                                       | 0.04                                     | 0.04                                     | 0(0)                                                   | 0.01(20)   | 0.03(80)  | 2.5/0.09                                                                   | 3.1/0.21           | 6.6/0.08; 13.0/0.06  | II–H3                   |
|                                        | 180       | 5                                       | 0.03                                     | 0.02                                     | 0(0)                                                   | 0(0)       | 0.03(100) | 2.5/0.10                                                                   | 3.0/0.12           | 6.3/0.05; 10.0/0.03  | II–H3                   |
|                                        | 180**     | 6                                       | 0.03                                     | 0.02                                     | 0(0)                                                   | 0(0)       | 0.03(100) | 2.5/0.19                                                                   | 3.5/0.11           | 5.0/0.06; 10.0/0.03  | II–H3                   |
| 190(8.0)                               | 15        | 184                                     | 0.35                                     | 0.37                                     | 0.02(5)                                                | 0.25(67)   | 0.10(28)  | 2.7/1.88                                                                   | 4.5/10.65          | –/–                  | II–H3*                  |
|                                        | 60        | 93                                      | 0.29                                     | 0.29                                     | 0.03(1)                                                | 0.13(44)   | 0.16(55)  | 2.7/0.26                                                                   | 3.7/1.28           | 6.3/1.85; 9.1/1.99   | II–H3                   |
|                                        | 90        | 45                                      | 0.16                                     | 0.16                                     | 0.02(1)                                                | 0.05(32)   | 0.11(67)  | 2.6/0.22                                                                   | 3.7/0.74           | 10.2/0.96            | II–H3                   |
|                                        | 120       | 13                                      | 0.05                                     | 0.05                                     | 0(0)                                                   | 0.01(23)   | 0.04(77)  | 2.6/0.19                                                                   | 3.7/0.28           | 7.3/0.12; 10.0/0.11  | II–H3                   |
|                                        | 150       | 7                                       | 0.03                                     | 0.03                                     | 0(0)                                                   | 0(0)       | 0.03(100) | 2.1/0.10                                                                   | 3.5/0.17           | 5.4/0.09; 10.0/0.05  | II–H3                   |
|                                        | 150**     | 8                                       | 0.03                                     | 0.03                                     | 0(0)                                                   | 0(0)       | 0.03(100) | 2.5/0.19                                                                   | 3.5/0.15; 4.4/0.10 | 8.1/0.07; 10.0/0.06  | II–H3                   |
| 190(6.0)                               | 30        | 184                                     | 0.36                                     | 0.39                                     | 0.03(7)                                                | 0.24(61)   | 0.13(32)  | 2.5/2.63                                                                   | 3.9/16.09          | –/–                  | II–H3*                  |
|                                        | 60        | 162                                     | 0.41                                     | 0.43                                     | 0.01(3)                                                | 0.29(68)   | 0.12(29)  | 2.5/1.28                                                                   | 4.2/5.95           | 5.5/6.64             | II–H3*                  |
|                                        | 90        | 108                                     | 0.32                                     | 0.34                                     | 0(0)                                                   | 0.19(57)   | 0.14(43)  | 2.7/0.56                                                                   | 3.7/1.56; 5.0/2.22 | 7.4/3.22             | II–H3*                  |
|                                        | 120       | 63                                      | 0.21                                     | 0.22                                     | 0(0)                                                   | 0.08(38)   | 0.14(62)  | 2.5/0.36                                                                   | 3.7/0.92           | 10.3/1.42            | II–H3                   |
|                                        | 150       | 30                                      | 0.10                                     | 0.11                                     | 0(0)                                                   | 0.03(35)   | 0.07(65)  | 2.7/0.32                                                                   | 3.7/0.69           | 9.0/0.45             | II–H3                   |
|                                        | 180       | 15                                      | 0.05                                     | 0.06                                     | 0.01(17)                                               | 0.01(17)   | 0.04(66)  | 2.5/0.29                                                                   | 3.7/0.33           | 9.0/0.12             | II–H3                   |

Table S2. *Cont.*

| Treatment Conditions                   |                 | $S_{\text{BET}}$ ,<br>$\text{m}^2/\text{g}$ | $V_{\text{BET}}$ ,<br>$\text{cm}^3/\text{g}$ | $V_{\text{BJH}}$ ,<br>$\text{cm}^3/\text{g}$ | Distribution of $V_{\text{BJH}}$ on $D_p$ (%) |          |          | $D_{\text{max}}/((dV/dD) \times 10^2)$ in Area of Pore Diameters, nm |           |          | Type of Hysteresis Loop |
|----------------------------------------|-----------------|---------------------------------------------|----------------------------------------------|----------------------------------------------|-----------------------------------------------|----------|----------|----------------------------------------------------------------------|-----------|----------|-------------------------|
| $T(\text{pH})$ ,<br>$^{\circ}\text{C}$ | $\tau$ ,<br>min |                                             |                                              |                                              | <3 nm                                         | 3–5 nm   | >5 nm    | 2.0–3.0                                                              | 3.1–5.0   | >5.0     |                         |
| 190(4.0)                               | 0               | 209                                         | 0.29                                         | 0.32                                         | 0.06(19)                                      | 0.20(62) | 0.06(19) | 2.0/2.96                                                             | 3.5/32.23 | –/–      | II–H3*                  |
|                                        | 30              | 213                                         | 0.33                                         | 0.36                                         | 0.05(13)                                      | 0.24(66) | 0.08(21) | 2.5/3.85                                                             | 3.7/28.08 | –/–      | II–H3*                  |
|                                        | 45              | 203                                         | 0.34                                         | 0.38                                         | 0.04(10)                                      | 0.25(67) | 0.09(23) | 2.5/3.13                                                             | 3.7/24.79 | –/–      | II–H3*                  |
|                                        | 60              | 203                                         | 0.35                                         | 0.38                                         | 0.03(8)                                       | 0.26(70) | 0.08(22) | 2.5/2.53                                                             | 3.7/20.0  | –/–      | II–H3*                  |
|                                        | 75              | 183                                         | 0.37                                         | 0.42                                         | 0.03(6)                                       | 0.29(70) | 0.10(24) | 2.7/2.70                                                             | 4.0/16.49 | –/–      | II–H3*                  |
|                                        | 90              | 200                                         | 0.39                                         | 0.42                                         | 0.02(5)                                       | 0.30(72) | 0.10(23) | 2.7/2.16                                                             | 4.2/14.29 | –/–      | II–H3*                  |
|                                        | 120             | 188                                         | 0.41                                         | 0.44                                         | 0.01(3)                                       | 0.32(73) | 0.11(24) | 2.7/1.33                                                             | 4.8/10.55 | –/–      | II–H3*                  |
|                                        | 150             | 178                                         | 0.43                                         | 0.46                                         | 0.01(2)                                       | 0.34(75) | 0.11(23) | 2.7/0.84                                                             | 4.7/8.94  | 7.5/4.80 | II–H3*                  |
|                                        | 180             | 155                                         | 0.42                                         | 0.44                                         | 0(0)                                          | 0.33(77) | 0.10(23) | –/–                                                                  | 3.9/2.54  | 6.3/6.50 | II–H3*                  |

\*: There is a definite site;  $\tau^{**}$ : after additional heating up to 400  $^{\circ}\text{C}$  (10  $^{\circ}\text{C}/\text{min}$ ).