SORTING SEMICONDUCTING SINGLE-WALLED CARBON NANOTUBES AND USING THEM AS RANDOM NETWORK OF FIELD-EFFECT TRANSISTORS

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Single-walled carbon nanotubes (SWCNTs) with incovalently attached iodine, were obtained by physical absorption. The different diameter sizes of SWCNTs, with different numbers of iodine molecule, enhance the density contrast between them which becomes evident in density gradient ultracentrifugation (DGU) targeted to sort certain species of SWCNTs. The results of optical absorbance and photoluminescence emission showed that iodine-assisted DGU preferentially separates semiconducting nanotubes with certain diameters [(6, 5), (7, 5), (8, 4), and (7, 6)]. We have applied these semiconducting, species enriched SWCNTs to prepare solution-processed field effect transistor (FET) devices with random nanotube network active channels. The devices exhibit stable p-type semiconductor behavior in air with very promising characteristics. The on-off current ratio reaches up to $2 \times 10^4$ within a narrow window of voltage ($-10$ V to 10 V), and estimated hole mobility of 21.7 cm$^2$ V$^{-1}$ s$^{-1}$.

Keywords: Carbon nanotubes; sorting; ultracentrifugation; transistor; mobility.

1. Introduction

Carbon materials, such as graphene, carbon nanotubes, ribbon and $C_{60}$ have attracted much research attention in recent years, especially for their applications in transistor, solar cell, capacitor, electrode field, etc. Among them, single-walled carbon nanotubes (SWCNTs) with different diameters and rotation angles have different intrinsic bandgaps; they can be divided into metallic/semi-conducting types, or armchair, zig-zag and chiral types. However, the coexistence of different types of species in as-production SWCNTs may cause problems in some real-life applications. For example, the metallic species in semiconducting SWCNTs field effect transistor (FET) clearly decrease the on-off ratio; the variation in species makes the
sensitivity low when they are used as photoluminescence sensors. These drawbacks make the species separation of SWCNTs becoming important. So far, certain powerful separation methods have been developed, such as DNA wrap-chromatography,\textsuperscript{11–13} density gradient ultracentrifugation (DGU),\textsuperscript{14–16} di-electrophoresis,\textsuperscript{17–19} agarose gel,\textsuperscript{20} selective chemistry, etc. Using the first or second method, only certain unique chiral species can be achieved. As for DNA wrap-chromatography method, the process is laborious and less efficient to get enough samples. Furthermore, the coating of DNA on the surface of nanotube is also a critical factor to acquire excellent electric properties. DGU process as a scale-up method (such as Nanointegrisce Inc) is promising as an elegant methodology in the SWCNTs separation fields. To our knowledge, this separation method depends on original types of raw materials. Arc discharge and laser vaporizing SWCNTs with less species and perfect carbon structures, are relatively easy to use in metallic/semiconducting separations. CoMoCAT and HiPco methods can generate more species; variations in contents of species are governed by carbon sources, catalysts and reaction conditions used in each method. This characteristic makes the separation effect less effective even if multi-color layers are present in the DGU process. The reason for this effect may be concluded as arising from density overlap of similar species of SWCNTs. Furthermore, different lengths of each species with similar diameters make this effect more pronounced. Recent papers have reported that a single nanotube existing in two or more chiral structures also makes it difficult to separate or sort single chiral species.\textsuperscript{29} Fortunately, Hersam’s group successfully used orthogonal iterative DGU to solve this problem.\textsuperscript{30} According to their findings, the ratio of sodium dodecyl sulfate (SDS) and sodium cholate (SC) is the key point in DGU process to achieve large density differences for SWCNTs species, especially for the similar diameter species. The present study was aimed at investigating other methods to reach similar separation goals.

In this study, we used iodine absorption in SWCNTs to boost DGU species separation. The heavy density of adsorbed molecular iodine on SWCNT increases the distance travelled by different species in DGU process, similar to the effect of SDS/SC co-surfactants. Through this modified DGU method, it is possible to obtain pure semiconducting species by using only SDS as surfactant. The separation results are also confirmed from the random network of FET.

2. Experimental Section

2.1. Materials

Chemicals and reagents were purchased from Aldrich and Acros Chemical Co., unless otherwise stated, and were used without further purification. All of the solvents were purified according to conventional methods when necessary. SWCNTs synthesized by the “HiPco” growth process\textsuperscript{31} were purchased from Unidym Inc (No. R0513). The nanotubes were used as purchased.

2.2. Measurements and characterization

UV-Vis–NIR absorption spectra were measured with a Shimadzu UV-3150 spectrophotometer. Photoluminescence excitation (PLE) spectroscopy measurements were conducted with a HORIBA Scientific Nanolog spectrofluorometer. The corresponding images of the SWCNT films were obtained by scanning electron microscopy (SEM, Hitachi S4800).

2.3. Iodine absorption by raw SWCNTs

To obtain iodine attached SWCNTs, 10 mg SWCNTs and a small iodine sphere (30 mg) were placed into a tube with a magnetic bar at 30°C for 1.5 h, 5 h and 10 h. After the absorption process, the corresponding absorption contents of iodine in SWCNTs, placed for different time periods, were 0.8 wt.%, 2.1 wt.% and 5.5 wt.% (the iodine concentration was determined by titration with sodium thiosulphate in KI solution). The SWCNTs samples in the corresponding samples were identified as I-08-SWCNTs, I-21-SWCNTs and I-55-SWCNTs. They were re-dispersed in 2 wt.% SDS for use in the DGU process.

2.4. Formation of density gradients

A density gradient column was formed in a polycarbonate centrifuge tube by layering, from top to bottom, 2.0 cm, 1.0 cm, 0.5 cm and 0.5 cm of 17.5%, 22.5%, 32.5%, and 40% w/v iodixanol, respectively. Each layer contained 2% w/v SDS. The SWCNTs
suspension (0.3 mL) was then carefully inserted on the top of this density gradient column. DGU was performed at 220 000 g for 15 h using a Hitachi ultracentrifuge with S52ST rotor. After centrifugation, colored layers were carefully extracted and collected using a micropipette.

2.5. Sample preparation for fluorescent quenching

The separated (6, 5) species was used as a sample in this part. After sorting process, the (6, 5) chiral solution obtained was characterized by UV absorbance at 750 nm and given a value 0.1, which corresponded to a SWCNT concentration of approximately 0.03 mg/L.32 It was adjusted to 0.01 mg/L by 2 wt.% SDS water solution before testing. The quenchers used were raw HiPco, raw arc discharge SWCNTs (from Carbon Solution Inc. No. 1309) and graphene (from Institute of Coal Chemistry, Chinese Academy of Science) in 2 wt.% SDS solution in water, with a concentration of 0.01 mg/L.

2.6. FET fabrication and characterization

For making SWCNTs FET devices, the DGU separated semiconducting SWCNTs solution above was filtered with Teflon filters (200 nm pore size). The SWCNTs solids were thoroughly washed by ethanol and re-dispersed in 1 wt.% SDS water solution. Subsequently, the concentration was adjusted by adding SDS surfactant solution until the UV-Vis-NIR absorbance of resultant solution at 750 nm was 0.2.32 Random networks of SWCNT thin film for FETs were fabricated by the spin coating method. The substrate used was a p-doped Si wafer coated with a layer of 300 nm SiO₂. The SWCNTs were randomly distributed on the substrate with high density. Arrays of metal source/drain (S/D) electrode pairs (channel was 5 μm long and 5 μm wide) were placed to establish contact of SWCNTs networks between each S/D pair. Electron-beam evaporation was used to define the S/D regions. The probing pads were gold/titanium (80 nm/2 nm). The samples were annealed at 220°C in an Ar atmosphere for better contact between the metal and the tubes. All electrical measurements were carried out in ambient environment using a Keithley semiconductor parameter analyzer model 4200-SCS.

The leakage of device from source and drain was controlled by using a wooden needle to scratch along the gold pads carefully. To improve the SWCNT film uniformity, the channel area was treated with O₂ plasma (100 W) for 3 min before application of the SWCNT dispersion. The treatment of device surfaces by O₂ plasma helps to limit the coffee-ring effect.33 In order to overcome the fringe effect of electrode pads, we use an analytical probe station with TV magnified image to line out a channel around the outside of source and drain pattern before characterization.

3. Results and Discussion

3.1. DGU separated colored layers

Figures 1(a)–1(d) show the separated images of pristine, I-08-SWCNTs, I-21-SWCNTs and I-55-SWCNTs samples after DGU process. When pristine HiPco SWCNTs dispersed in SDS were used directly for DGU, the semiconducting and metallic nanotubes had small differences in density and hence, could not be well separated. Figure 1(a) shows that there were approximately three layers after DGU process: the top thin layer was violet-blue, the middle was deep green, and the bottom layer was red-black. These three layers had fuzzy interfaces and some overlaps; this means low efficiency of separation of coexisting multi-species or semiconducting/metallic nanotubes. In order to overcome this problem, we used iodine absorption in SWCNTs to increase the corresponding density of species so that chiral SWCNTs could be sorted more effectively during the DGU process, as shown in Figs. 1(b)–1(d). This key treatment was effective in

Fig. 1. Sorting of HiPco SWNTs (n, m) structures by using iodine assisted DGU with 15 h at 22 000 g. Images of centrifuge tubes: (a) Pristine; (b) I-08-SWNTs; (c) I-21-SWNTs and (d) I-55-SWNTs; (e) the distinct four colored bands in enlarged I-21-SWNTs image are layers enriched as f1–f4 fractions (color online).
making overlap of species disappear in the DGU step for different diameters of SWCNTs having different of absorption of iodine contents. Figure 1(e) shows the enlarged image of sample I-21-SWCNTs after DGU process. Four layers (f1–f4) from top to bottom: violet, blue, green and red layers are clearly visible now. Figure 1(d) shows that use of higher levels of iodine in the SWCNT led to increased distance between different colored layers; however the enriched content of each layer also decreased correspondingly. Comparison of the four samples in Figs. 1(a)–1(d) showed that the sediments on the bottom also increased with the iodine-treatment time. Furthermore, the lower colored bands, with heavy density SWCNTs species, moved closer to the bottom of centrifuge tube. For example, the reddish layer in Fig. 1(c) had dropped down to a deeper location in Fig. 1(d). These phenomena clearly show that the level of iodine absorption in SWCNT has a critical effect on separation of species in DGU process.

### 3.2. Optical spectroscopy

In order to verify the detailed species separation results of DGU, PLE maps of pristine and the four separated fractions (f1–f4) of I-21-SWCNTs are shown in Fig. 2. For pristine SWCNT, which is a mixture of several species, the chiral indices \((n, m)\) for the identified tubes are labeled in Fig. 2(a). The assignments are based on fitting the energy bandgaps from the first \((E_{11}^S)\) and second \((E_{22}^S)\) van Hove transitions to the emission and excitation energies, respectively, using formulas derived from four-parameter tight binding models.\(^{34,35}\) Comparing the PLE peak positions with positions for the corresponding \((n, m)\) species reported in Bachilo et al.,\(^{36}\) reveals a red shift of approximately 1–2% for all samples. This is due to the effect of the surrounding SDS on the local dielectric environment. As reported earlier,\(^{37,38}\) the quantum PL efficiency for each chirality of tube is different. The abundance of individual species was determined from the peak height above the baseline divided by a theoretical intensity factor.\(^{39}\) The suspension species of pristine are summarized in the graphene sheet map (Fig. 3) and Table 1.

<table>
<thead>
<tr>
<th>Species</th>
<th>Chiral Angles</th>
</tr>
</thead>
<tbody>
<tr>
<td>(6, 5)</td>
<td>6.95° for (6, 5) to 8.95° for (10, 2)</td>
</tr>
</tbody>
</table>
Fig. 2. (a) PLE maps of pristine SWNTs; (b) absorbance spectra of sorted layers, with inserted image of sorted layers in vial; (c) PLE maps of f1 fraction; (d) f2 fraction; (e) f3 fraction; (f) f4 fraction.
enrichment performance of the nonlinear gradient, the method described by Ghosh et al.\textsuperscript{16} was used. The near-infrared absorption spectra of the four separated fractions were fitted, as shown in Fig. 2(b), by superimpositions of Voigt functions centered at the E\textsubscript{11} peaks of specific \((n, m)\) species. Assuming equal oscillator strengths for these transitions, and neglecting the possible presence of metallic species, the \((n, m)\) purity of each fraction was computed as the ratio of the area of the dominant peak to the sum of the peak areas (Supplementary Fig. S3). The calculated purity of the species for \((6, 5)\), \((7, 5)\), \((8, 4)\) and \((7, 6)\) are 91.2\%, 71.2\%, 83.2\% and 55.6\% respectively.

3.3. Fluorescent quenching of chiral SWCNTs

The sorted chiral SWCNTs fractions, as obtained by DGU, have high stabilities. For example, the \((6, 5)\) species solution (Fig. S4 in supporting information) was analyzed once a week after the sorting process was finished. After 35 days of storage, the five plots obtained were almost identical, which suggested that the SWCNTs were individually dispersed even after long storage time. This excellent property is important for chiral SWCNTs to be used as chemosensors. In real-life applications, SWCNTs are perfect solutions for eliminating background noise, thus providing high sensitivity to analyzing minute traces of analytes.

Usually, the fluorescence quenching behavior can be simply described by the Stern–Volmer equation\textsuperscript{40}:

\[
F_0/F = 1 + K_{sv}[Q],
\]

where \(F_0\) is the fluorescence intensity in the absence of quencher, \(F\) is the fluorescence intensity in the presence of quencher, \([Q]\) is the quencher concentration, and \(K_{sv}\) is the Stern–Volmer constant. When we use this equation to obtain the linear quenching curve, it is important to control experimental factors such as oxygen, SWCNTs concentration, temperature and pH value. Therefore, the concentrations of the chiral SWCNTs and quencher system were reduced in order to obtain linear Stern–Volmer plot, as shown in Fig. 5. Furthermore, low concentrations of the SWCNTs solution increase the quenching efficiency and provide a high entropic driving force in an analytical system.\textsuperscript{41}

Individual dispersion of \((6, 5)\) semiconducting SWCNT (0.01 mg/L in 2\% by wt. SDS water solution) had one fluorescence peak at 990 nm, with an intensity of 3500, when it was irradiated at 570 nm. The changes in fluorescence of the SWCNT, after addition of pristine HiPco, Arc and graphene, are shown in Fig. 5(a)–5(c). The most obvious quenching effect was induced by graphene.

<table>
<thead>
<tr>
<th>((n, m))</th>
<th>Diameter (nm)</th>
<th>Chiral angle (deg)</th>
<th>Calculated intensity\textsuperscript{a}</th>
<th>PL peak intensity</th>
<th>Calibrated PL peak intensity\textsuperscript{b}</th>
<th>Calibrated content (%)</th>
</tr>
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<tbody>
<tr>
<td>(6, 5)</td>
<td>0.757</td>
<td>27.02</td>
<td>0.67</td>
<td>721</td>
<td>1076</td>
<td>15.4</td>
</tr>
<tr>
<td>(7, 5)</td>
<td>0.829</td>
<td>24.50</td>
<td>0.71</td>
<td>1166</td>
<td>1642</td>
<td>23.5</td>
</tr>
<tr>
<td>(7, 6)</td>
<td>0.895</td>
<td>27.46</td>
<td>0.47</td>
<td>618</td>
<td>1315</td>
<td>18.8</td>
</tr>
<tr>
<td>(8, 3)</td>
<td>0.782</td>
<td>15.03</td>
<td>2.13</td>
<td>627</td>
<td>294</td>
<td>4.2</td>
</tr>
<tr>
<td>(8, 4)</td>
<td>0.840</td>
<td>19.11</td>
<td>0.46</td>
<td>854</td>
<td>1857</td>
<td>26.6</td>
</tr>
<tr>
<td>(9, 4)</td>
<td>0.916</td>
<td>17.48</td>
<td>0.70</td>
<td>418</td>
<td>597</td>
<td>8.5</td>
</tr>
<tr>
<td>(10, 2)</td>
<td>0.884</td>
<td>8.95</td>
<td>2.38</td>
<td>480</td>
<td>202</td>
<td>3.0</td>
</tr>
</tbody>
</table>

\textsuperscript{a}Calculated intensity: calculated PL intensity from Oyama et al. \textsuperscript{39}  
\textsuperscript{b}Calibrated PL peak intensity: PL peak intensity/calculated intensity.
Fig. 4. (a) Density profiles of the DGU medium before DGU and after 15 h of DGU at 220,000 g. Densities were deduced from refractive index measurements (see Supplementary Information); (b) Densities of the SDS-suspended SWNT species as a function of SWNT diameter.

Fig. 5. (a) Quenching behaviors of sorted (6, 5) SWNTs by pristine HiPco; (b) Arc and (c) graphene. The Stern–Volmer constants are shown in (d): pristine HiPco (1), Arc (2) and graphene (3).
followed by Arc and HiPco, the order depending upon the quenching mechanism. Decrease in fluorescence of SWCNT can be caused by several possible effects: energy-transfer or electron-transfer caused by bundling, presence of fluorescence-suppressing impurities such as acid or oxygen, changes in absorption (spectral bleaching), and quenching by either static or dynamic mechanisms. In our system, bundling is probably the main factor affecting fluorescence intensity. As shown in Fig. 5(d), graphene has large surface area and high pi-pi interaction with SWCNT (6, 5), resulting in relatively easy formation of aggregates such as graphene/SWCNTs bundling. This type of structure decreases the fluorescence intensity by energy-transfer or electron-transfer processes, with the Stern–Volmer constant, $K_{sv}$ having highest value (2.01 × 10^6 g L\(^{-1}\)). Compared with graphene, Arc and HiPco type SWCNTs have weak abilities to form bundling structures. Their $K_{sv}$ constants are 7.06 × 10^3 g L\(^{-1}\) and 1.97 × 10^5 g L\(^{-1}\), respectively. Arc type SWCNT has larger average diameter than HiPco type SWCNT, which suggests that Arc SWCNT has higher $K_{sv}$ value than of HiPco type. The sorting chiral SWCNTs are also promising candidates as fluorescence biosensors. This potential is currently being investigated in our lab.

### 3.4. SWCNT-based FETs

The strongest evidence of semiconducting nanotube enrichment is the application of enriched SWCNTs as random networks in thin film FETs. Significant contamination with metallic SWCNTs results in conducting pathways through the SWCNT network, thus lowering the on-off ratio.

Network FETs were made by spin-coating nanotube solution into FET channel. The geometry of the resulting fabricated bottom-gated FETs is shown in Fig. 6. After enrichment process by iodine-assisted DGU, the separated chiral species were filtered and washed many times to clean the impurities; subsequently, trace amounts of iodine were further discarded in a vacuum box maintained at 50°C for overnight. SWCNT solution droplets were spun onto the channel area until the resistance of the device was less than ~1 MΩ. The SWCNT network in the channel was characterized by SEM. Figure 6(b) shows the SWCNT network in the device channel. The measured density of nanotubes in the channel area was about 20–30 tubes μm\(^{-2}\).

Figure 6(c) shows typical transfer relationship of the forward sweep of drain current ($I_d$) versus gate voltage ($V_g$) for a bottom-gated FET made with enriched four chiral SWCNTs; the devices for (6, 5), (8, 4), (7, 5) and (7, 6) exhibit typical p-type behavior and have effective carrier mobility of 21.7, 4.3, 1.3, 0.1 cm²V\(^{-1}\)s\(^{-1}\) and on-off ratio of 2 × 10^4, 4 × 10^2, 1 × 10^3, 60 respectively. Figure 6(d) shows the spatial map of individual device on a 5 × 10 array. The different colors represent the on-off ratio of each device. Compared with pristine HiPco, which has a very low on-off ratio (in the range of 1 to 10) as measured in our previous study, the devices, prepared as described above from sorted SWCNTs, exhibit high values of on-off ratio and effective mobility. Moreover, it was found that very sparse films (1 to 4 SWCNT μm\(^{-2}\)) do not form sufficiently conductive paths to yield measurable on-state current. On the other hand, increasing SWCNTs density to build electrical paths for devices will improve the possibility of creating connecting paths of metallic SWCNTs, in channels which limit increases in the on-off ratio. Owing to greater ease in sorting the four species of SWCNTs by iodine-assisted DGU, it is possible to increase the density (thickness) of the networks without building up excessive metallic SWCNT conduction paths. The on-currents increases in this order: from 0.03, 0.5, 1 to 11 μA for (7, 6), (7, 5), (8, 4) and (6, 5) respectively. The on-off ratio shows similar trend. These results imply the presence of some metallic SWCNTs in (7, 6), (7, 5) fractions although the content of metallic SWCNT in these samples was quite low. Also, these results demonstrate that it is possible to tune the mobility of solution-processed SWCNT transistors by controlling the density of the SWCNT films.

In our previous research, we had found that the residual impurity on the surface of sorted SWCNTs increases tube/tube contacts but reduces carrier mobility, thus limiting the maximum current and lowering the on-off ratio. This problem can be eliminated by cleaning the surface of SWCNTs. In the present study, we report that spinning a PMMA thin film on SWCNTs network increases the performance of the device. Without PMMA film, devices from the four species of SWCNTs clearly show hysteresis on the $I_d$–$V_g$ curves [Fig. 7(a)]; these are attributed to the existence of impurities such as H₂O, as reported by other researchers. Our data confirm these results, and shows that the properties
Fig. 6. (a) Schematics of FET geometry; (b) the SEM image of (6, 5) SWNT networks in the device channel; (c) $I_d-V_g$ curves of FET devices of four sorted fractions with polymethylmethacrylate (PMMA) coating; (d) the spatial color maps of on-off ratios of sorted fractions.

Fig. 7. (a) $I_d-V_g$ cycle curves of FET devices for four sorted fractions without PMMA coating; (b) effect of post treatment processes on FET property.
imparted to the films are not easily destroyed by washing the surfactant or by heating during the vacuum process. It is evident from Fig. 7(b) that coating a thin layer of PMMA on the SWCNTs device can decrease the hysteresis. This is presumably due to blockage of water absorption path on SWCNTs surface, which keeps the SWCNTs networks from getting wet in air. Furthermore, Fig. 7(b) also suggests that the PMMA coating process increases the on-current. When we tested the device without the coating process, it was noted that the forward and backward sweep of \( I_d-V_g \) curves were unable to connect with each other (the end point being higher than start point in Fig. 7(b), filled dotted line). This result suggests that some changes occur in the SWCNTs channel; it seems likely that at least one of these changes is shrinkage of the SWCNTs network during testing the first time (input current). The shrinking of networks results in increase in diameter of the electrical pathways, thus yielding higher end current. This situation becomes more significant after testing is done several times. In contrast, PMMA film coating restricts the shrinking movement of SWCNTs network, resulting in stable \( I_d-V_g \) cycles, even after testing many times.

4. Conclusions

In this study, significant amount of \( (n, m) \) selective enrichment was performed by introducing both nonlinear DGU and physical iodine absorption for HiPco SWCNTs. Optical absorbance and photoluminescence emission revealed that appropriate iodine absorbance enhances the density contrast between different chiral species, thus assisting DGU in preferentially separating semiconducting nanotubes with certain diameters i.e., \( (6, 5), (7, 5), (8, 4) \) and \( (7, 6) \). Furthermore, the purity of single chirality SWCNTs can reach up to 91.2%, with high stability characteristics. FET fabricated from solutions enriched with semiconducting species showed excellent device characteristics. Different chiral species of SWCNT showed significant differences in device performance. We believe that further understanding and refinement of this separation method can lead to higher purity of single chirality SWCNTs and improved device performance. Also, the as-prepared SWNT could be applied in PN heterojunction solar cell and severed as photoactive materials in polymer-based solar cells.

Acknowledgments

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References

Supporting Information

Estimation of device mobility

The effective field-effect mobility is estimated by:

\[ \mu = 10^4 \times \frac{I_d}{V_{gs}} \times \frac{L}{W} \times \frac{1}{C_{ox} V_{ds}}. \]

Here, \( C_{ox} \) is determined by \( \varepsilon_0 \varepsilon_r A/d \), where \( \varepsilon_0 \) is permittivity of free space \( (8.85 \times 10^{-12}) \), \( \varepsilon_r \) is relative permittivity \( (3.9) \), \( A \) is unit area and \( d \) is the gate silicon oxide thickness \( (3 \times 10^{-7}) \). \( L \) and \( W \) represent the channel length and width, respectively.

Fig. S1. Density and corresponding iodixanol concentration as a function of refractive index (two red dot lines are error ranges) (color online).

Fig. S2. Absorbance spectra of sorted layers (f1–f4).

Fig. S3. The calculated purity of each sorted fraction.
Fig. S4. The stability of sorted (6, 5) fraction after 35 days (testing frequency is once per week).