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Effect of Pluronic block copolymers on aqueous dispersions of graphene oxide

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1 **Abstract:** The effect of Pluronic block copolymer F127 and P123 on aqueous
2 dispersions of graphene oxide (GO) was studied. The combination of optical
3 microscope, rheometer, scanning electron microscopy and X-ray diffractometer
4 studies indicated that the introduction of Plurionic F127 and P123 could markedly
5 change the dispersion, flow and processing behaviors of GO in water. Interestingly,
6 the ordered-lamellar-structure composite tapes were easily fabricated by casting the
7 GO/F127 dispersions through a doctor blade method while it was not obtained by
8 casting the GO/P123 dispersions. The study on the intercalation structure of GO
9 composite tapes suggested that the ordered lamellar structure of GO/F127 composite
10 tapes originated from the anchor-buoy-type adsorption of F127 on the GO surface. In
11 the anchor-buoy-type configuration, long ethylene oxide (EO) segments of F127 were
12 extended out into water to form a hydration protection layer, which effectively
13 prevented the aggregation of GO sheets. Instead, the protective effect was weaker for
14 P123 due to its very short EO segments, and the phase-separated dispersions were
15 present with higher GO content.

16

17 **Keywords:** Pluronic block copolymer; graphene oxide; dispersion; intercalation
18 structure; adsorption behavior

1. Introduction

Graphene oxide (GO) is usually made by reacting graphite powder with strong oxidants.¹ After oxidation, the single-atom-thick GO sheets are exfoliated, bearing hydroxyl and epoxide functional groups on their basal planes, in addition to carbonyl and carboxyl groups located at the sheet edges. The presence of these functional groups makes GO sheets strongly hydrophilic, which allows them to readily swell and disperse in common polar solvents including water. This solution process of GO thus offers a practical and facile route to prepare graphene-based composites, paper, or thin film.² GO sheets can be viewed as a two-dimensional polymer molecule that behave like surfactants-being enriched at interfaces, lowering the interfacial energies, and dispersing carbon nanotubes in water.³ This suggests an amphiphilic surface, since a single layer is essentially complete surface. Therefore, the modification of such a surface would have a direct impact on a broad range of technological applications of this material.

Much effort has been put into chemically exfoliating or intercalating modification for graphite in solution phase to obtain colloidal suspensions of graphene. And some success has been achieved for the mass production of graphene.⁴ The next huge challenge is the large-scale assembly of the exfoliated graphene sheets into a desired format, such as thin films and papers. Current methods for fabricating structure-controllable graphene films or papers are limited to batch processes, which is just suited for applications at the lab scale. For example, layered graphene films or papers have been assembled layer-by-layer from monolayers formed in a

23 Langmuir-Blodgett trough⁵ or made by filtering graphene suspensions across a porous
24 membrane.⁶ They were not suitable for fabricating a graphene film with sufficient size
25 for testing, evaluation and use in a device.

26 In this present work, Pluronic block copolymers F127 and P123 were exploited
27 to modify the surfaces properties of GO. In water for Pluronics F127 and P123, the
28 hydrophobic propylene oxide (PO) blocks tend to be adsorbed on particle surfaces and
29 the hydrophilic ethylene oxide (EO) blocks extend into aqueous solution and provide
30 steric stabilization to the particles.⁷ Hence, it is of both scientific curiosity and
31 technical importance to know how these atomically thin sheets of GO behave when
32 interacting with amphiphilic polymers. Once the behaviors of GO aqueous dispersions
33 could be controlled by adding amphiphilic polymers, this would significantly broaden
34 the applications of this material.

35

36 **2. Experimental**

37 Exfoliated GO platelets (purity > 99 %, single layer ratio ~ 99 %, and average
38 aspect ratio ~1600) were purchased from Nanjing XFnano (China) and used as
39 received. The Pluronic block copolymers F127 (ethylene oxide₁₀₆-propylene
40 oxide₇₀-ethylene oxide₁₀₆, $M_w = 12\ 600$, the value of critical micelle concentration
41 (CMC) in water at 25 °C is 1.0 g/L) and P123 (ethylene oxide₂₀-propylene
42 oxide₇₀-ethylene oxide₂₀, $M_w = 5\ 800$, the value of CMC in water at 25 °C is 0.052 g/L)
43 were obtained from Sigma-Aldrich and used as received.

44 Appropriate amounts of GO were dispersed individually in deionized water (18

45 $M\Omega \cdot \text{cm}$) by sonication for 1 h to form GO aqueous suspensions. Then, the GO/
46 amphiphilic copolymer dispersions were prepared by dissolving the Pluronic block
47 copolymers into the above GO suspensions under gentle agitation, and the solutions
48 were sonicated for 30 min. The formulations of all the GO dispersions were
49 summarized in Table 1, which FG and PG were referred to the use of Pluronic F127
50 and P123, respectively. Note that the concentrations of amphiphilic copolymers were
51 higher than their critical micellar concentration for all the GO/amphiphilic copolymer
52 dispersions. All the GO dispersions were casted on the clean glass plates using a
53 DS1000 automatic control coater (Dream Science Co., Korea) with a 1.0 mm blade
54 gap. The coating speed was 50 mm/s. The tapes were dried at 25 °C for several days
55 and then removed from the glass templates to obtain large, free-standing tapes. The
56 thickness of these tapes was controlled by the casting times and the gap between the
57 blade and substrate.

58 All the dispersions were rested at 25 °C for two weeks before the tests. Optical
59 microscopy images were obtained using a Nikon digital measuring microscope
60 (MM-400/L, Japan) equipped with a Nikon microscope digital camera and E-Max
61 software. Rheological tests were performed on a Rheometer (Physica MCR 301,
62 Anton Paar Germany GmbH) equipped with 50 mm diameter cone plates set 0.054
63 mm apart. All the measurements were done at 25 °C using a temperature controlled
64 water bath. The relationships of viscosity vs shear rate were obtained from the mode
65 of steady flow. The morphologies of the composites were investigated by field
66 emission scanning electron microscope (Hitachi S-4800, Japan) at 10 kV acceleration

67 voltages. Sampling X-ray diffraction (XRD) patterns were obtained using a Rigaku
68 D/Max-2000 X-ray diffractometer (CuK α radiation at 1.54 Å) at 40kV and 100 mA.

69

70 **3. Results and discussion**

71 **3.1 Optical study**

72 Experiments to study the effect of Pluronic block copolymers F127 and P123 on
73 aqueous dispersions of Graphene Oxide (GO) were performed. As presented in Fig. 1,
74 these aqueous dispersions exhibited different appearances with naked eyes after they
75 were prepared by mild sonication. For the GO solution with lower concentration, after
76 mild sonication, GO-01 and FG-11 were reddish brown and transparent fluids, which
77 suggested a homogeneous dispersion of GO in water. However, for PG-11, it showed
78 the inhomogeneous, reddish brown and milk-like appearance. The similar
79 chocolate-milk-like appearance was also observed for the higher GO concentration
80 samples, except for PG-13 and PG-15 (which showed a turbid appearance). Note that
81 an inhomogeneous, chocolate-milk-like appearance could be mistaken for the
82 aggregation of GO. But, in fact, it was suggestive of a nematic liquid crystal. On the
83 contrary, PG-13 and PG-15 showed the phase-separated state with the turbid
84 appearance after they rested in one month (Fig. 1), which was due to the aggregation
85 of GO.

86 The optical microscopy images of GO and GO/amphiphilic copolymer
87 dispersions (located between a pair of crossed polarizers, where the birefringent
88 optical texture reflects the local orientation of the GO platelets) were showed in Fig. 2.

89 A typical nematic schlieren texture consisting of dark and bright brushes was
90 observed in GO-05. However, such a texture was not present in GO-01 and GO-03.
91 Our observation was in agreement with predictions from Onsager's theory,⁸ which
92 gave a critical volume fraction of 0.25 %. Recently, Kim *et al.*⁹ and Xu *et al.*¹⁰ also
93 reported that the nematic state was formed in GO aqueous dispersions when GO
94 contents were higher than the critical concentrations (depending on the aspect ratios
95 of GO platelets). For PG-13 and PG-15, the nematic schlieren texture was absent and
96 just small bright spots were observed under the crossed polarizers, which indicated
97 that they had the locally ordered structure. However, the nematic texture was
98 observed in FG-13 and FG-15 although more disclinations were present.

99 **3.2 Rheological study**

100 The flow curves of GO and GO/amphiphilic copolymer dispersions were showed
101 in Fig. 3. From the rotational-shear rheological data (viscosity vs. shear rate) of
102 aqueous GO dispersions (Fig. 3a), GO-05 exhibited typical shear flows characterized
103 by decreasing viscosity upon shearing, but GO-01 and GO-03 did a simple fluid.
104 Strong shear thinning of GO-05 might be attributed to the liquid crystal state where
105 the anisotropic sheets were prone to align in a shear field. Interestingly, three distinct
106 regions were observed in FG-15 and PG-15 (Fig. 3b): shear-thinning at high shear
107 rates, a plateau with approximately constant viscosity at intermediate rates, and an
108 additional regime of shear thinning at low rates. Such a behavior was also found in a
109 variety of liquid crystalline polymers.¹¹ Generally, it was believed to reflect a piled
110 polydomain texture composed of nematic domains, which possess little or no

111 macroscopic flow-induced orientation. This was supported by our optical images
112 above.

113 **3.3 XRD and SEM study**

114 Here GO composite tapes were fabricated by casting the above GO dispersions
115 through a slit by the motion of a doctor blade relative to a substrate. Compared to
116 Langmuir-Blodgett and filtration methods,^{2,12} the casting method circumvented the
117 limitations of these small-scale batch processes, and produced large-area GO
118 composite tapes. During the casting process, colloidal suspensions need to flow easily
119 through the gap between the blade and substrate under shear, and maintain the shape
120 and thickness of the cast tape once shearing is removed. Such a behavior is typically
121 achieved by casting shear thinning suspensions. Here the concentrated GO dispersions
122 exhibited the strong shear thinning behavior (Fig. 3), thus being adequate for effective
123 casting.

124 Interestingly, a layered structure was observed in the cross-section of the dried
125 tapes from the castings of GO-05 and FG-15, with parallel striations to the tape
126 surfaces (Fig. 4a and 4b), while the structure was lost for PG-15 (Fig. 4c), where the
127 cross-section appeared more isotropic. The anisotropy was caused most likely by the
128 alignment of the GO sheets.

129 The XRD patterns of GO and its composite tapes were showed in Fig. 4d. All the
130 GO tapes exhibited a characteristic diffraction peak around 10.74° (2θ),
131 corresponding to the layer distance of 0.84 nm. From the studies on water content of
132 the GO galleries, Medhekar et al.¹³ pointed out that the thickness of GO slab was ca.

133 0.60 nm and the size of water was ca. 0.28 nm. The measured layer distance (0.84 nm)
134 therefore could be attributed to approximately one water molecule-thick layer that is
135 presumably hydrogen-bonded between the GO sheets (ca. 0.60 nm + 0.28 nm). For all
136 the FG tapes, the diffraction peaks shifted to 5.98° (2θ , the layer distance of 1.48 nm),
137 which indicated that they presented the intercalated structures. Interestingly, the PG
138 tapes showed variable layer distances, the layer distance of 1.33 nm (2θ , 6.70°) for
139 PG-15, 1.58 and 3.15 nm (2θ , 5.60° and 2.80°) for PG-13, and 3.15 nm (2θ , 2.80°)
140 for PG-11. Moreover, their diffraction peaks were unusually weak, compared to that
141 of the FG and GO series. The low intensity of the basal reflections from the
142 distributed GO platelets should be suggestive of a disordered stacking or exfoliation.

143 From the studies on the adsorption of PEO based surfactants in the clay galleries,
144 Deng et al.¹⁴ pointed that the thickness of monolayer with planar-conformation was ca.
145 0.4 nm. Harris et al.¹⁵ studied the conformation of PEO intercalated in clay and MoS₂
146 through two-dimensional double-quantum NMR. They found that the layer distance
147 was ca. 1.36 nm ($\Delta h = 0.73$ nm) for the intercalation of a monolayer with planar
148 conformation and ca. 1.56 nm ($\Delta h = 0.93$ nm) for the intercalation of a monolayer not
149 with planar conformation. Therefore the small layer distance suggested a single-layer
150 arrangement with nonplanar-conformation for the FG series (1.48 nm) and PG-13
151 (1.58 nm) and a single-layer arrangement with planar-conformation for PG-15 (1.33
152 nm), while the large layer distance (3.15 nm for PG-11 and PG-13) suggested a
153 multi-layer arrangement in the GO galleries. The above XRD results confirmed the
154 intercalation of F127 and P123 in the graphene galleries.

155 3.4 Adsorption behaviors

156 Recently, Pluronic block copolymers were demonstrated to disperse carbon
157 nanotubes¹⁶⁻¹⁸ and graphene/graphene oxide¹⁹⁻²² in aqueous solutions. In their works,
158 the combination of experimental observations, computer simulations and theoretical
159 analysis showed that the hydrophobic PO blocks tended to be attached at the
160 nanotube/graphene surface, whereas the hydrophilic EO blocks were extended out
161 into the aqueous phase, providing a steric barrier. Observation of our results suggested
162 that such a protection mechanism also happened in our dispersions. However, the
163 differences between our Pluronic F127 and Pluronic P123 systems showed that the
164 length of hydrophilic EO blocks had strongly influenced on dispersion and solution
165 behaviors of GO.

166 Given the intercalation structure with single-layer form (1.48 nm) for the FG
167 composite tapes, we expected F127 molecules to be adsorbed on the GO surface with
168 the anchor-buoy-type structure in the suspensions (Fig. 5): the PO blocks formed a
169 collapsed, relatively thin layer (the anchor) which were strongly adsorbed on the GO
170 surface, and the EO blocks formed the adjacent swollen layer (the buoy), also called
171 the “brush” configuration. Previously, many researches were carried out on
172 intercalation and adsorption of PEO and its derivatives in the clay galleries.^{15,23-25}
173 They found that such a “brush” adsorption behavior of PEO and its derivatives often
174 led to the intercalation of a monolayer (the layer distance ca. 1.36-1.56 nm) or a
175 bilayer (the layer distance ca. 1.68-1.77 nm) in the clay galleries¹⁵, depending on the
176 adsorption amount of modifiers.

177 For the PG composite tapes, the intercalation structure with single-layer form
178 (1.33 nm for PG-15, 1.58 nm for PG-13) and multi-layer form (3.15 nm for PG-13
179 and PG-11) should be attributable to different adsorption mode. The single-layer
180 intercalation arose from the anchor-buoy-type adsorption of P123 molecules on the
181 GO surface in the dispersions. However, the multi-layer intercalation was thought to
182 be from the adsorption of P123 with clusters on the GO surface (Fig. 5). Brandani and
183 Stroeve observed the presence of micellar-like aggregates (also clusters) at the surface
184 for P103 (ethylene oxide₁₇-propylene oxide₆₀-ethylene oxide₁₇) through AFM.²³

185 The interaction between colloidal particles determines their colloidal stability.²⁶
186 GO is known to form a colloidal solution in water due to electrostatic repulsion
187 between the ionized carboxylic groups. When two GO sheets approach each other,
188 they experience both electrostatic repulsion and van der Waals attraction. Note that
189 the scaling law of van der Waals potential (W) versus separation (d) strongly depends
190 on the geometry of the interacting bodies: $W \sim 1/d^6$ for two points, $W \sim 1/d^5$ for two
191 parallel chains, and $W \sim 1/d^2$ for two parallel planes.²⁶ Therefore their van der Waals
192 potential rapidly decays as the separation increases if two sheets are brought together
193 in an edge-to-edge geometry. To the contrary, their van der Waals potential slowly
194 decays when the sheets are brought together in a face-to-face geometry. Therefore, the
195 GO colloidal solutions would be unstable during storage due to the long-range
196 face-to-face attraction, which was indeed in agreement with our observation.

197 In our work, the introduction of Pluronic F127 and P123 provided a steric barrier
198 through the extension of the hydrophilic EO blocks into the aqueous phase to hinder

199 the long-range face-to-face attraction. However, we observed that all the PG
200 dispersions were more unstable during storage (Fig.1). Recently, Seo et al.
201 investigated the dispersion efficiency for a set of 14 different Pluronic block
202 copolymers through optical absorbance measurements.¹⁹ They found one principal
203 trend that graphene were exfoliated more efficiently as the length of the EO block
204 increased. Their work revealed that Pluronic block copolymers having short PEO
205 segments did not provide sufficient steric hindrance to prevent graphene sheets from
206 interacting and ultimately aggregating with one another in solutions. Therefore such
207 instability in all the PG dispersions was due to short EO block of P123. The
208 aggregation thereby prevented the formation of ordered lamellar structure during
209 drying the PG dispersions. In aqueous environments, even short hydrophilic EO
210 blocks (ca. 20 repeat units) could not shield efficiently from the long hydrophobic PO
211 blocks (ca. 70 repeat units) when P123 molecules were attached on the GO surface
212 through the “brush” configuration. As a result, the hydrophobic PO block attached on
213 the GO surface continued to adsorb the excess of P123 in the aqueous bulk phase, and
214 finally formed the micellar-like aggregates (also clusters) at the surface.

215 To the contrary, the EO block of F127 was enough long. According to the
216 literature reported,²⁴ the adsorption of F127 on the clay surface could form a
217 hydration protection layer with 3.5 nm thick in the aqueous solution. The hydration
218 protection layer with such thickness had a certain repulsion, which could effectively
219 prevent the aggregation of nano-particles. And so the stable dispersions were got for
220 FG serials observed in Fig. 1. Furthermore, such a stable repulsion protective effect

221 was conducive to the formation of ordered lamellar structure during drying, because it
222 could prevent the formation of agglomerated gel with disordered structures. Based on
223 the above discussion, the schematic on the adsorption state of F127 and P123 on the
224 GO surface was provided with different modes for adsorption behavior as shown in
225 Fig. 5.

226

227 **4. Conclusions**

228 In summary, the effect of Pluronic block copolymers F127 and P123 on aqueous
229 dispersions of GO was investigated. FG-13 and FG-15 showed the nematic texture
230 although the present disclinations were more than GO-05. However, PG-13 and
231 PG-15 showed the phase-separated state after one month. Moreover, all the casted FG
232 tapes presented the intercalated structures (the layer distance of 1.48 nm), but the PG
233 tapes showed variable layer distances, the layer distance of 1.33 nm for PG-15, 1.58
234 and 3.15 nm for PG-13, and 3.15 nm for PG-11. Importantly, the ordered lamellar
235 structure was easily obtained by casting the FG-15 dispersions through a doctor blade
236 method while it was not obtained by casting the PG-15 dispersions. These differences
237 in the behaviors were due to different adsorption mode of F127 and P123 on the GO
238 surface in the suspensions: the anchor-buoy-type adsorption of F127 which led to
239 the single-layer intercalation, and the presence of “anchor-buoy-type brush” and
240 clusters of P123 on the GO surface which resulted in the variable intercalation. In the
241 anchor-buoy-type configuration, long EO segments of F127 were extended out into
242 water to form a hydration protection layer, which effectively prevented the

243 aggregation of GO sheets. And so the stable dispersions were got and the
244 ordered-lamellar-structure tapes were easily casted for FG serials. For the adsorption
245 of P123 on GO surface, the protective effect was weakened due to the very short EO
246 segments. And so the phase-separated dispersions were present with higher content of
247 GO.

248

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254

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Figure Caption

Fig.1. Photos of GO/ amphiphilic copolymer dispersions

Fig.2. Optical microscopy images of GO and GO/ amphiphilic copolymer dispersions located between a pair of crossed polarizers

Fig.3. Flow curves of GO and GO/ amphiphilic copolymer dispersions

Fig.4. SEM images of (a) GO-05, (b) FG-15, (c) PG-15 and (d) XRD spectra of GO and its composite tapes

Fig.5. Models for adsorption behaviors of Pluronics F127 and P123 on the GO surface

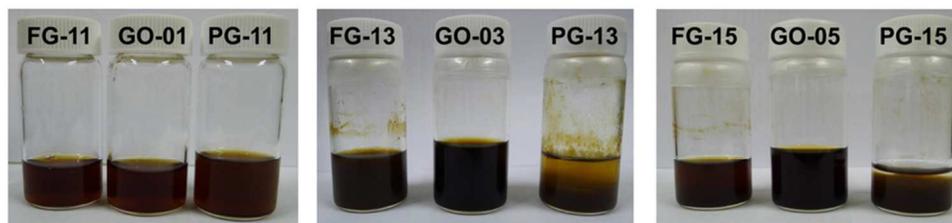


Fig.1. Photos of GO/ amphiphilic copolymer dispersions
40x9mm (600 x 600 DPI)

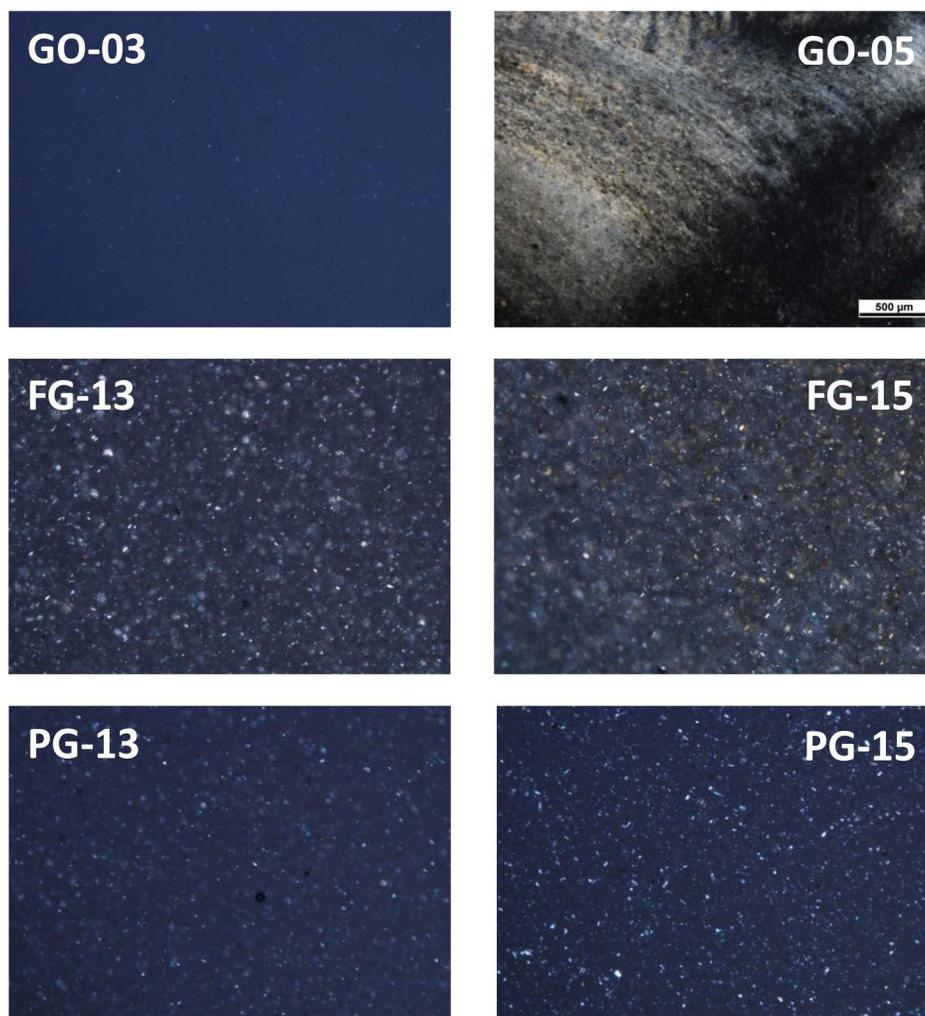


Fig.2. Optical microscopy images of GO and GO/ amphiphilic copolymer dispersions located between a pair of crossed polarizers
120x132mm (600 x 600 DPI)

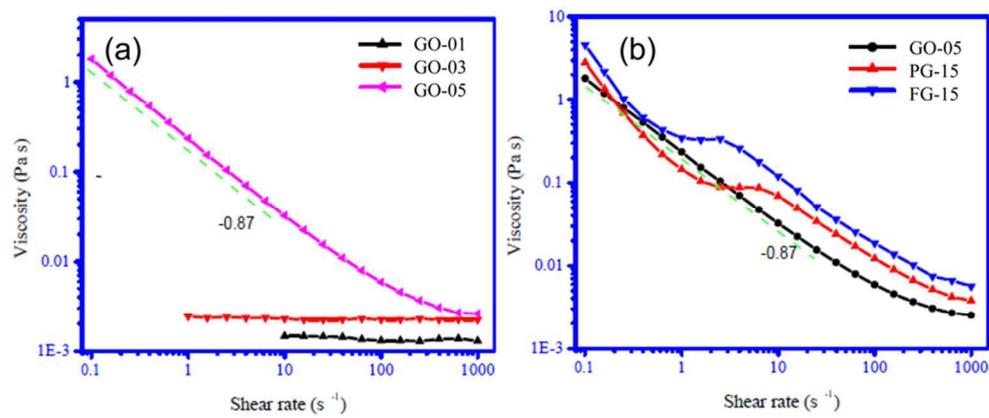


Fig.3. Flow curves of GO and GO/ amphiphilic copolymer dispersions
40x17mm (600 x 600 DPI)

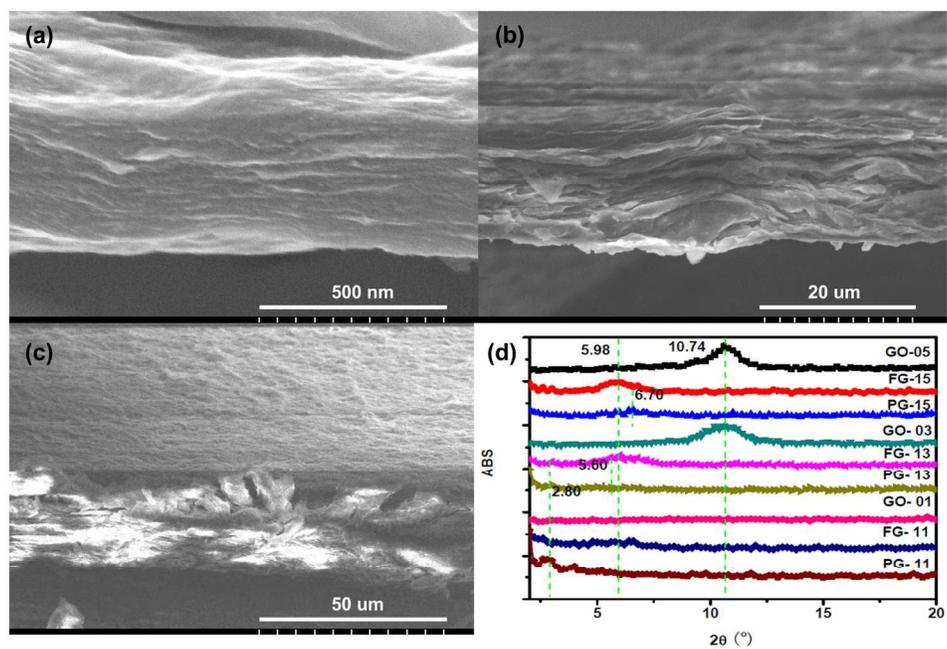


Fig.4. SEM images of (a) GO-05, (b) FG-15, (c) PG-15 and (d) XRD spectra of GO and its composite tapes 80x53mm (600 x 600 DPI)

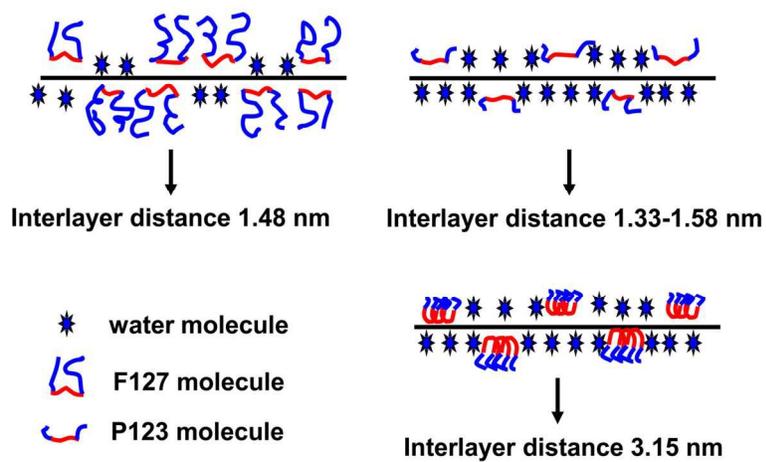


Fig.5. Models for adsorption behaviors of Pluronic F127 and P123 on the GO surface
80x59mm (600 x 600 DPI)