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研究課題名(和文) Hierarchical liquid crystal assemblies based on large graphene oxide sheets and nonionic organic compounds

研究課題名(英文) Hierarchical liquid crystal assemblies based on large graphene oxide sheets and nonionic organic compounds

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研究成果の概要(和文)：このプロジェクトでは、非イオン性界面活性剤によって形成されるミセル、金属クラスター、金属酸化物ナノシートなどのさまざまなコロイドを酸化グラフェンに関連付け、主にエントロピー効果によって駆動されるさまざまなコロイド集合体を理解します。コロイド構造の安定化と階層集合体の形成の可能性をもたらす推進力の理解というこの研究プロジェクトの基本的な関心を越えて、GOはメソポーラス材料や機能性材料の調製のためのテンプレートシステムまたは支持相として使用されることに成功しました。金属クラスターおよび金属酸化物ナノシートとの結合/組み合わせを通じて、それぞれ殺菌剤および電気化学的用途に使用されます。

研究成果の学術的意義や社会的意義

電気化学および殺菌フィルム用途の製造の範囲において、当社はGOと他のコロイドの結合によって形成されるナノ複合材料を開発しました。得られたナノ複合材料は、短いTIS2を含めることによってGOの再積層が制限されるため、GOの電気容量が最大25%向上することが明らかになりました。支持相として使用される酸化グラフェンは、Moナノクラスターで均一に修飾することができ、グラム陽性菌(黄色ブドウ球菌)を除去するための殺菌剤用途で反応性酸化種を生成するナノ複合材料を生成します。

研究成果の概要(英文)：The main objectives of the JSPS Kakenhi C project were to associate different colloids: micelles formed by nonionic surfactants, metal clusters and metal oxide nanosheets to graphene oxides and understand the different colloidal assemblies mainly driven by entropic effects. Beyond the fundamental interests of this research project about the understanding of the driving forces leading to the stabilization of the colloidal edifices and possible formation of hierarchical assemblies, GO was successfully used as a template system or supporting phase for the preparation of mesoporous materials and functional materials through their association / combination with metal clusters and metal oxide nanosheets for bactericide and electrochemical applications respectively.

研究分野：materials science, colloids

キーワード：colloids nanocomposites graphene oxide surfactants mesoporous materials

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Overview and main results of the research project:

The main objectives of the JSPS Kakenhi C project were to associate different colloids: micelles formed by nonionic surfactants, metal clusters and metal oxide nanosheets to graphene oxides and understand the different colloidal assemblies mainly driven by entropic effects. Beyond the fundamental interests of this research project about the understanding of the driving forces leading to the stabilization of the colloidal edifices and possible formation of hierarchical assemblies, GO was successfully used as a template system or supporting phase for the preparation of mesoporous materials and functional materials through their association / combination with metal clusters and metal oxide nanosheets for bactericide and electrochemical applications respectively.

1. Graphene oxide nanosheets

Graphene, the emblem of 2D nanomaterials, consists of a single sp^2 carbon layer showing unique properties (different from those of the initial layered graphite material it originates from): (i) a large specific surface ($> 2000 \text{ m}^2 \text{ g}^{-1}$), (ii) excellent electrical conductivity characteristics to that of a semiconductor without any band gap and (iii) very good mechanical properties. Initially synthesized through the etching and oxidation of graphite to prepare graphene after a reduction process (thermal, UV radiation...), graphene oxide (GO) nanosheets exhibit as well outstanding properties that tackle the interests of both the scientific and industrial communities. The inclusion of oxygen within the C sheets modulates the electrical properties with the generation of novel hybrid orbitals. In addition, the oxidation of C sheets confers a hydrophilic behavior of the GO nanosheets making them dispersible in aqueous media that can self-organize in nematic liquid crystalline phase, used a template to produce C fibers as well as supporting phase for further colloids: clusters, nanoparticles of various dimension to prepare nanocomposite materials.

On their surfaces, GO nanosheets show several functional groups: ketone, carboxyl, aldehyde, hydroxyl... as well as graphene like patches preserved from any alteration, nor chemical changes, representing as many reaction sites as possible for future reactions and/or functionalization operations for the grafting of organic compounds or their association to other colloids. The oxidation rate of GO, that can be adjusted following the concentration of the oxidant during the synthesis or through a reduction process of the nanosheets, determines its reactivity as well as the band gap and the possible photocatalytic properties. The presence of the diverse functional groups on GO allows covalent bonding with nanoparticles and/or other colloids. While giving sustainable nanocomposites, this association may show the disadvantages to affect the GO surface reactivity and its electronic properties depending on the grafted sites. Preparation of nanocomposites can be driven through non-covalent processes involving electrostatic interactions, hydrogen bonds, van der Waals and π - π interaction between the associated nanoparticles and GO of which structure and its properties are preserved.

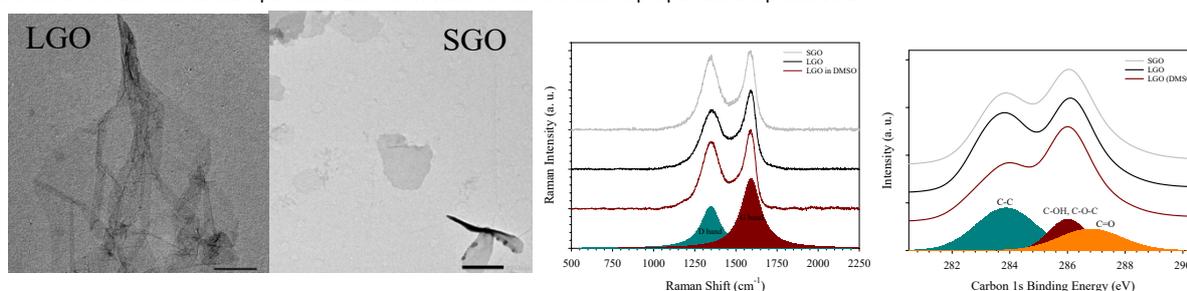


Figure 1: Characterization by transmission electron microscopy of the 2 types of synthetic graphene oxides (SGO and LGO) showing short (about 200 nm) and large ($>10 \mu\text{m}$) lateral sizes. Characterization, by Raman scattering (a) and XPS (b), of the 2 types of synthetic graphene oxides (SGO and LGO) showing short (about 200 nm) and large ($>10 \mu\text{m}$) lateral sizes while displaying equivalent oxidation rate but different number of physical defects with shorter graphene patch like zone for the case of SGO.

The graphene oxide (GO) nanosheets were prepared by using the modified Hummer's method, of which details are published elsewhere. This classic route of synthesis of GO based on the oxidation and exfoliation of the graphite led to large 2D nanomaterials, reminiscent of the initial precursor geometry, showing a lateral size of about several ten micrometers (20-40 micrometer). The diversity of the chemical landscape of OG nanosheets and their surfactant character, highlighted when using 2D nanomaterials as emulsion stabilizers (Pickering type emulsions) allow them to be dispersible in many polar and non-polar organic solvents as demonstrated by several studies. Using ultrasonic treatment at high power during several minutes, the lateral size of GO can be drastically reduced, down to 200 nm. In addition, in collaboration ship with Prof. Sugahara from Waseda University, the chemical nature of GO can be easily changed through thermal reduction or using cationic surfactants as chemical modifiers leading to a hydrophobic surface or bearing cationic charges respectively.

2. Liquid crystalline assemblies formed by nonionic surfactants

Surfactants are amphiphilic molecules which contain a hydrophilic head and a hydrophobic tail. In contact with water, those molecules align are organized at the interface with water by showing their hydrophilic heads in water and the hydrophobic tails in the air to minimize their free energies. As the concentration of surfactants increases, they may cover the water-air interface, and above a certain temperature (Kraft point) and a critical micelle concentration (CMC), surfactants self-assemble in micelles. As the concentration continues to rise, the micelles formed by the amphiphilic molecules change in both size and shape depending on the interactions with their surroundings, and can self-assemble in conventional spherical micelles, and lyotropic liquid crystalline (LC) phases: bicontinuous cubic, hexagonal, and lamellar phases.

Amphiphilic alkylpoly (ethylene oxide) (C_nE_m) nonionic surfactants are known to form various lyotropic LC phases depending on their molecular structure, composition, temperature, and concentration in water.

The temperature and concentration phase diagrams of the $C_{12}E_{5-8}$ -H₂O systems that could be determined by several complementary techniques: polarized optical microscopy (POM), small angle X-ray scattering (SAXS), small angle neutron scattering (SANS) in collaboration with Dr. Hiroki Iwase in the frame of experiments at J-Parc neutron facilities done March 2021, and near infrared spectroscopy in collaboration with Dr. Jelena Muncan from Kobe University are shown in Figure 2.

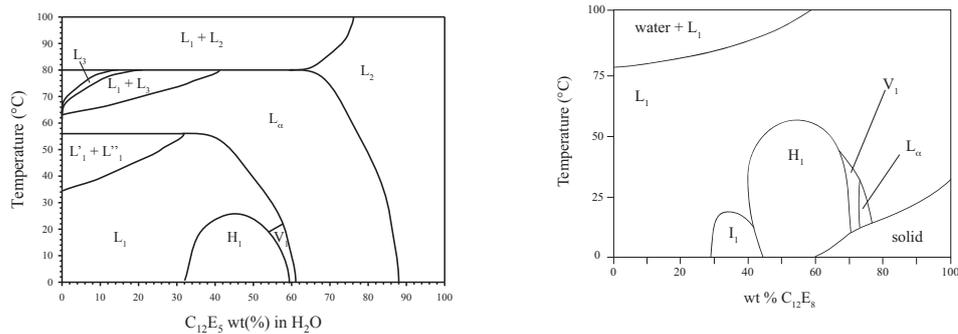


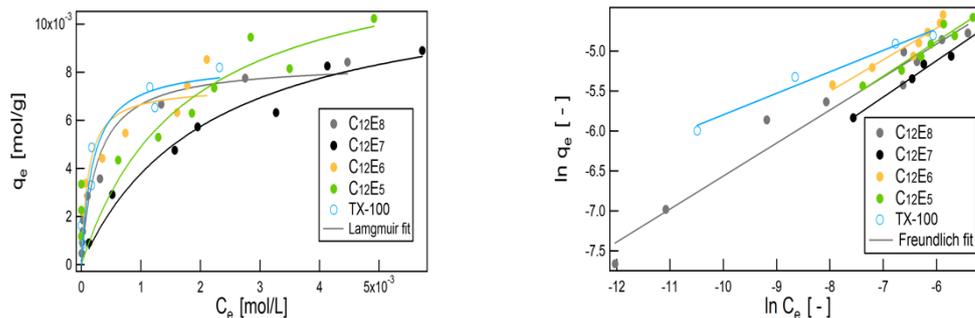
Figure 2: Phase diagrams of the $C_{12}E_5$ and $C_{12}E_8$ -H₂O systems. L_1 stands for cylindrical micelle phase, H_1 , the hexagonal phase, V_1 the cubic phase, L_α the lamellar phase, L_3 the sponge phase and L_2 the reverse micelle phase.

3. Association of GO to polyethylene glycol alkyl ether (C_nE_m) nonionic surfactants

3.1. Low affinity of the nonionic surfactants with GO

The affinity of a series of polyethylene glycol alkyl ether (C_nE_m) nonionic surfactants, self-assembling in diverse lyotropic liquid crystalline phases (hexagonal, lamellar, sponge phases) that can be used as template systems for the synthesis of mesoporous materials, with graphene oxide (GO) nanosheets was investigated through the determination of adsorption isotherms, by changing the hydrophilic lipophilic balance (HLB) parameter of pentaethylene glycol monododecyl ether ($C_{12}E_5$), hexaethylene glycol monododecyl ether ($C_{12}E_6$), heptaethylene glycol monododecyl ether ($C_{12}E_7$) and octaethylene glycol monododecyl ether ($C_{12}E_8$) and nonionic surfactants.

The adsorption isotherms were obtained through total organic carbon analyses allowing one to determine the concentrations of the surfactants before and after being into contact with GO and were fitted by Langmuir and Freundlich models. Langmuir equation is mainly valid if the molecules are adsorbed in a monolayer on the adsorbent surface, while Freundlich model is an empirical model ($q_e = K_F C_e^{1/n}$, where q_e is the adsorbed amount, C_e stands for the equilibrium concentration, K_F : the Freundlich constant) considering the non-equivalence of the adsorption sites and the diversity of the interaction mechanisms leading to the adsorption. Thus, due to the diversity of the adsorption sites and heterogeneity of GO, Langmuir model appears less suitable than empirical the Freundlich one a proper fitting of the adsorption data. The adsorbed amounts of nonionic surfactants showing large HLB values ($C_{12}E_8$ and TX-100) increased gradually as the equilibrium concentration was increased and exhibited a saturation or plateau at high concentration regime, while for the other surfactants, no saturation of the adsorption was observed.



Figures 3-4: Adsorption isotherms at the equilibrium of a series of polyethylene glycol alkyl ether (C_nE_m) nonionic surfactants: pentaethylene glycol monododecyl ether ($C_{12}E_5$), hexaethylene glycol monododecyl ether ($C_{12}E_6$), heptaethylene glycol monododecyl ether ($C_{12}E_7$) octaethylene glycol monododecyl ether ($C_{12}E_8$) and octylphenol ethoxylate (TX-100). Fitting of the adsorption isotherms using Freundlich model.

From the collected adsorption isotherms and the fitting parameters, the affinity between GO and nonionic surfactants appeared quite low. From the r^2 values (not shown) quantifying the agreement between the fitting curves and the adsorption data, the whole adsorption isotherms showed a better agreement between the experimental data and the adsorption model equations by using Freundlich than Langmuir one. This is because Langmuir model is valid for monolayer adsorption of adsorbate molecules at adsorption sites on the adsorbent surface and is based on the theory that all adsorption points are equivalent and that leading force for the adsorption remains constant. Since each of GO sheet and surfactant has multiple and different adsorption sites involving weak molecular interaction mechanisms: H-bonds, ion-dipole interaction, and Van der Waals interaction, the Freundlich empirical model seemed to be the most appropriate adsorption model to adjust the experimental data, with $n > 1$ indicating the adsorption was favorable while showing low or moderate values for the Freundlich constants: 0.04-0.1 L g⁻¹.

If the surfactants were adsorbed onto GO surface involving the hydrophilic head group of surfactants and the oxygen moieties of GO, the adsorbed amounts of nonionic surfactants should be increased as the HLB value was increased. However, according to raw adsorption data, the fitting parameters and the Freundlich constant K_F which are very low, the tendency was inverted. This indicates that surfactants may be adsorbed with other interaction mechanisms onto GO surface. Even if it is not possible to exclude the possible adsorption onto the hydrophilic

moieties, it is likely that the main driving forces leading to the adsorption may occur through the hydrophobic moieties by involving Van der Waals forces as it could be observed for graphite substrate. The resulting aggregates formed onto graphite surface display hemi-micelle structure, reminiscent to those of the bulk surfactant. Therefore, since GO display numerous C sp² patches that were not altered during its synthesis of (oxidation and exfoliation of graphite), it is likely that nonionic surfactants may form aggregates onto carbon plane as it could be observed and confirmed by molecular dynamics calculations for the adsorption of C₁₂E₈ onto graphite surface. Even if further experiments or calculations are required to confirm the main adsorption mechanisms and we cannot exclude neither the possible adsorption of nonionic surfactants onto the hydrophilic moieties, the adsorption isotherms and fitting parameters stress out the importance of the HLB parameter about the adsorbed amounts and affinity of the nonionic surfactants with GO. Surfactants with large HLB seemed to be poorly adsorbed with the possibility to even reach a saturation of the adsorption with the observation of a plateau for TX-100 and C₁₂E₈. In contrast, the adsorption for surfactants showing low HLB (e.g. a more hydrophobic character) was favored with possible occupation of the C plane of GO through Van der Waals and formation of hemi-micelles that can then facilitate the adsorption of further nonionic molecules through other mechanisms. The shorter the hydrophilic head group chains was, the more number of surfactant molecules was packed in hemi cylindrical structure because of steric hindrance of hydrophilic head group.

3.2. Impact of GO onto the morphology of the C_nE_m nonionic surfactants micelles

As colloidal systems, GO and the micelles formed by nonionic surfactants, are ruled by excluded volume effect. The latter represents the volume inaccessible to the other colloids, leading to their stacking in well-organized structure to increase the entropy that will be larger in such configuration than in randomly fashion at the same colloidal volume fraction. The excluded volume effects are the cause of the formation of liquid crystalline assemblies of GO at low concentration of the nanosheets as well as being responsible of the stacking of the micelles made of nonionic surfactants in hexagonal fashion and other organizations (cubic, lamellar...) at large volume fraction of surfactants.

If the universality of volume effects allows one to describe various disorder-order phase transitions for one type colloids, those effects are more difficult to apprehend for bi-colloidal systems, where the nature, morphology, geometry, size, and concentration of colloids are all important parameters to consider for the stability of resulting edifices. In the case of a dispersion with rigid inorganic GO and surfactants forming micelles of different size and morphology, whose systems somehow resemble as living polymers with constant destructuring and restructuring of the micelles, the understanding on the impact and role of excluded volume effects turn out to be much more delicate and challenging.

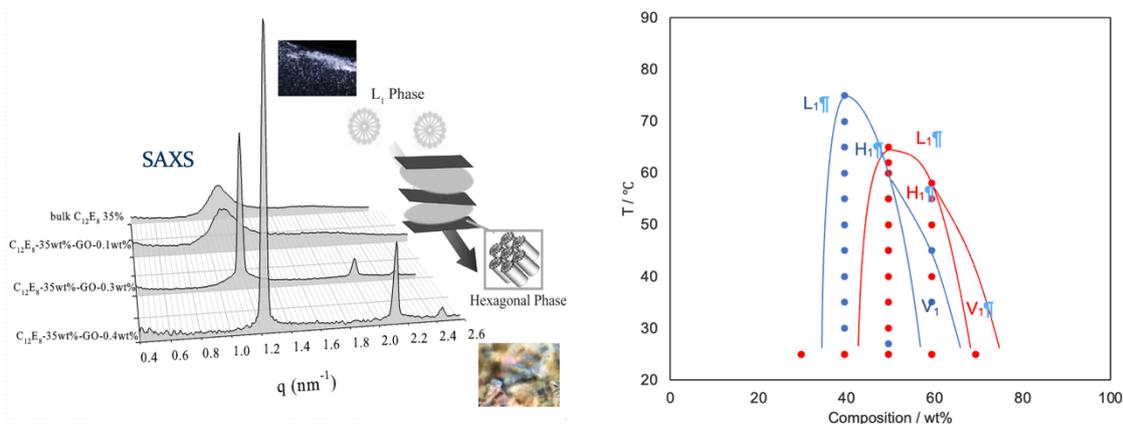


Figure 5: SAXS profiles of bulk C₁₂E₈ nonionic surfactant at the volume fraction of 35% in water and in the presence of GO of which lateral size reaches 100 micrometers at the concentration of 0.1, 0.3 and 0.4 wt%. Phase diagrams of the bulk C₁₂E₈-H₂O system (red) and that of C₁₂E₈-H₂O in the presence of GO of which lateral size was about 1-10 micrometers (blue).

As a consequence of excluded volume effects of the inorganic nanosheets as well as the poor affinity of the nonionic surfactants to GO, their inclusion to the aqueous surfactant solutions led to a reorganization of the morphology of the C₁₂E₈ surfactant with the observation of a hexagonal phase (H₁ phase) at 40 wt% where only a micellar phase (L₁ phase) was expected. Interestingly, this shift in concentration for the formation of the hexagonal phase and stacking of the cylindrical micelles in hexagonal fashion was even more pronounced as the lateral size was larger (Figure 5). To note that no hexagonal phase was formed in the presence of small GO of which lateral size was about 200 nm introduced at the same density in the colloidal dispersion. This confirmed that the lateral size of the GO sheets and their derivative excluded volumes represents an important parameter in the reorganization of the surfactants. Here, the excluded volume should be understood as a volume of the nanosheets including a certain amount of water since the nanosheets were already solvated (with a slight change in the surfactant concentration), that cannot participate in the formation of the micelles expected from the bulk C₁₂E₈-H₂O phase diagram. Furthermore, nanosheets in solution are believed to be mainly planar or flat, but T. Nakato *et al.* revealed that only 40% of them are flat and the others are rounded (35%), folded (14%), undulated (11%). These changes of morphology (that cannot occur in the case of small GO) may also affect considerably the volume of water in close interaction with GO surface, reducing the number of free molecules that can participate in the formation of the H₁ phases. Indeed, comparing the *d* values of bulk C₁₂E₈ sample (50 wt%) and C₁₂E₈-GO samples (50 wt% C₁₂E₈ and 0.8 wt% GO dispersion) as shown in 3.2., the values decreased in the order: bulk C₁₂E₈, C₁₂E₈-SGO, C₁₂E₈-LGO. It confirms that the large sheets trap more water molecules than the small sheets and reduces the amount of free water molecules, leading to tightly packed surfactant molecules. Thus, this can explain why large nanosheets at low concentration induce a phase change by reducing the amount of free (or demobilized) water molecules that can be associated with the C₁₂E₈.

By using shorter lateral size GO (lateral size estimated at about 1-10 micrometers), this shift in concentration induced by excluded volume effects was also observed at large concentration regime of the surfactants with the formation of a bicontinuous cubic phase (V₁ phase) at 60 wt% where normally the V₁ phase was expected to be formed at 70 wt% for the bulk C₁₂E₈-H₂O system (Figure 8). Thus, the formation of the H₁ phases at lower concentration underlined the lack of adhesive forces or interactions between GO sheets and C₁₂E₈ surfactants due to the

ionized GO surface and the presence of hydronium ions and corroborated the previous adsorption isotherms at the equilibrium pointing out the poor affinity or cohesion of the nonionic surfactants to GO.

Figure 8:

Indeed, with carboxylic groups decorating GO, the resulting dispersions of the mixture of GO and C₁₂E₈ exhibited a pH below the value of 3, without nevertheless affecting the formation of the nonionic surfactant micelles which are pH independent. At such pH of 2-3, the Debye screening length can be easily determined and estimated at $\kappa = 0.304I^{-1/2} \approx 3 \text{ nm}$ with I the concentration of the hydronium ions ($I=10^{-\text{pH}}$). As it could be pointed out by the determination of the adsorption isotherms, within this length that needs to be considered for carboxylic groups and other hydrophilic organic moieties decorating GO surface, it is unlikely to find C₁₂E₈ molecules, nor their micelles, but mobilizes many water and hydronium ions. Although the colloidal mixtures appear to be stable without any phase separation, local segregation may be possible (modulated by the Debye screening length). It leads to a decrease of the amount of water molecules (or increase of the local concentration of the surfactant) inducing a phase reorganization of the C₁₂E₈-H₂O system. Interestingly, in addition to the observation of a shift in concentration for the whole phase transitions that are driven by excluded volume effects or other entropic effects, the presence of GO enlarges the domains in temperature of both hexagonal and cubic phases, with the observation of an enlargement of more than degrees for several concentrations of surfactant. Here, the close association of water surrounding GO may act as a hermetic cage or confinement matrixes that reduce the mobility or dynamics of the micelles. Indeed, nonionic surfactants can be seen as living polymers with micelles continuously deconstructing and reconstructing themselves. The introduction of nanoparticles may lead to a slowing down of the dynamics or a reduction of the frequency of the deconstructing-reconstructing micelle formation, by keeping the morphology of the micelles in larger time scale as well as an enlargement of their temperature domains. Previously, the addition of laponite (clay mineral) or aeorosil silica beads at low concentration regime in membrane systems was recognized to enlarge their temperature phase domains, by stabilizing the interaction between the bilayers and thus enlarged the temperature phase domains. Here, GO shows a moderate affinity to the nonionic surfactants, but its presence seems to stabilize the difference membrane phases leading to a close packing of the micelles at lower concentration than the bulk systems while enlarging their temperature phase domains.

3.3. Synthesis of mesoporous materials based on GO-C₁₂E₈ nanocomposites

Surfactants which are amphiphilic molecules self-assemble in various lyotropic liquid crystalline phases: lamellar, hexagonal and cubic phase by minimizing their free energy in a solvent. These organic arrangements and in especially the hexagonal phase can be used as a template for the synthesis of silica based mesoporous materials. Their association with GO induces to a phase reorganization and a close packing of the cylindrical micelles in a hexagonal fashion. Thus, the presence of large nanosheets contributes to further structuration of the systems with hierarchical advanced organization at several levels (between the sheets as well as at the scale of the cylindrical micelles).

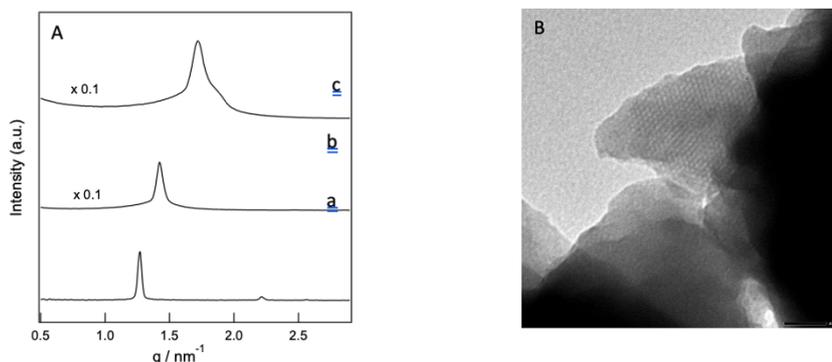


Figure 6: (A) SAXS profiles of the sample: C₁₂E₈-GO sample (50 wt% C₁₂E₈ and 0.8 wt% GO dispersion) in the process of synthesizing porous materials (a) before adding TMOS, (b) before calcination and (c) after calcination. (B) TEM observations of the resulting mesoporous material exhibiting a larger specific surface area than that made by bulk C₁₂E₈, due to the presence of GO.

SAXS profiles showed different reflections characteristics of the hexagonal phase (H₁) formed by the C₁₂E₈ in the presence and GO, confirming the conservation of the hexagonal lattice after the addition of tetramethyl ortho siloxane (TMOS) as well as the calcination process (Figure 6). The decrease in d value during the synthesis process results from a dehydration condensation. The characterization of the resulting mesoporous materials by Raman scattering revealed two bands (G and D bands) characteristics of GO confirming that the inorganic nanosheets were through the whole synthesis procedure. While displaying regular pores, the BET results confirmed the formation of mesoporous materials displaying a large specific surface of about 490 and 550 m² g⁻¹ for bulk C₁₂E₈-H₂O system and GO-C₁₂E₈. The slight increase of about 13% of the specific area in the case of the hybrid porous materials comes from the presence of GO of which existence was confirmed by Raman scattering. Nevertheless, while not being clearly observed from TEM observations, the association of GO did not affect the formation of the mesopores which appeared to be well in a row regularly. The presence of the inorganic nanosheets did not induce a second order porous structure that may be responsible of the enlargement of the specific surface area, which here can be explained by just the presence of GO, characterized by its very large specific surface area.

4. Further prospects of a project for the preparation of nanocomposites based on GO

In the scope of producing electrochemical and bactericide film applications, we developed nanocomposites based on the association 2D GO nanomaterials and other nanosheets (titanium disulfide, niobate) as well as metal nanoparticles (Mo₆ clusters). After a careful understanding of the resulting microstructures formed by the association of the colloids, the nanocomposites including graphene oxide and titanium disulfide nanosheets revealed an improvement up to 25% of the electrical capacitance of GO due to the limitation of restacking of GO by the inclusion of short TiS₂ and improvement of the resulting specific surface areas. Graphene oxide used as supporting phase, can be uniformly decorated with Mo nanoclusters leading to nanocomposites producing reactive oxidative species with bactericide applications for the elimination of gram-positive bacteria (staphylococcus aureus).

5. 主な発表論文等

〔雑誌論文〕 計0件

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1. 発表者名 R. Guegan
2. 発表標題 Hybrid Materials based on colloidal association of inorganic and organic phases showing functional properties
3. 学会等名 Chemical Wisdom Colloquium, Waseda University (招待講演)
4. 発表年 2021年

1. 発表者名 R. Guegan
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3. 学会等名 Alumni of the Department of Applied Chemistry of Waseda University (招待講演)
4. 発表年 2021年

1. 発表者名 H. Ramezani, R. Guegan, Y. Sugahara
2. 発表標題 Realistic modeling of graphene oxide (GO) nanosheets at a molecular level
3. 学会等名 Zaiken Meeting
4. 発表年 2021年

1. 発表者名 M. Tanimura, R. Chisoku, S. Yoshiyuki, R. Guegan
2. 発表標題 Contribution of large graphene oxide sheets to a phase reorganization of nonionic surfactant
3. 学会等名 The 68th JSAP Spring Meeting
4. 発表年 2021年

1. 発表者名 M. Tanimura, R. Chisoku, S. Yoshiyuki, R. Guegan
2. 発表標題 Contribution of large graphene oxide sheets to a phase reorganization of nonionic surfactant
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4. 発表年 2021年

1. 発表者名 X. Huang, R. Guegan, Y. Sugahara
2. 発表標題 Phase reorganization of a nonionic surfactant in the presence of large graphene oxides
3. 学会等名 Zaiken Festa
4. 発表年 2021年

〔図書〕 計0件

〔産業財産権〕

〔その他〕

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6. 研究組織

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7. 科研費を使用して開催した国際研究集会

〔国際研究集会〕 計0件

8 . 本研究に関連して実施した国際共同研究の実施状況

共同研究相手国	相手方研究機関
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