

Article

Soak-and-Squeeze Deposition of Graphene Oxide on Open-Cell Foams for Detergent Removal

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Abstract

This study investigates the deposition of graphene oxide (GO) coatings on open-cell polyurethane foams through a soak-and-squeeze process for detergent removal. GO dispersions between 0.25 and 4 g/L were rheologically characterised to relate apparent viscosity to coating deposition. A first rationalisation of the squeeze-coating process was proposed by estimating the characteristic shear rates during squeezing, obtaining values of 12.2 s^{-1} and 50.4 s^{-1} for 20 and 60 PPI foams, respectively. Multiple deposition cycles were performed, achieving GO loads up to 12.7 wt% for both types of foam. The adsorption behaviour of GO powder toward a commercial detergent solution containing linear alkylbenzene sulphonate (LAS) was investigated at 25, 40 and 60 °C. UV-Vis spectroscopy was used to monitor concentration changes, while conductivity analyses investigated the temperature-dependent aggregation behaviour of the detergent system. The adsorption kinetics were well described by the Elovich model, suggesting adsorption on heterogeneous GO surfaces. Filtration tests using GO-coated foams showed a decrease in conductivity during flow-through experiments, whereas UV-Vis measurements indicated limited LAS removal under dynamic conditions, likely due to insufficient contact time between the detergent and the coated surface. Static contact tests performed for 1 h at 25 °C showed a reproducible detergent concentration decrease of approximately 16–18%. This value was further increased up to 65% under optimised conditions by modifying the GO-to-detergent ratio and foam configuration. The results demonstrate that the soak-and-squeeze approach is a simple and effective method for producing reproducible GO coatings on open-cell foams, while indicating that sufficient contact time is required to promote detergent removal.

Keywords: graphene oxide; open-cell foam; soak-and-squeeze coating; coating; viscosity; adsorption; detergent removal



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1. Introduction

In recent years, water has emerged as an increasingly critical resource, driven by the combined effect of climate change, population growth and industrial and agricultural demands. Globally, freshwater withdrawals are mainly driven by agriculture, which accounts for about 72% of total withdrawals, followed by industry and domestic use, with municipal demand increasing mainly in rapidly developing urban areas. The withdrawals affect the water stress situation, which is defined as the availability of water with respect to its demand [1]. In addition, water pollution represents a global concern, decreasing the availability of water and contributing to the water stress situation. Water bodies can

be contaminated by different classes of pollutants, including emerging pollutants, such as pharmaceuticals, microplastics, PFAS and detergents and surfactants [2,3]. These are relevant contaminants because they are widely used in domestic and industrial activities and can enter aquatic environments through wastewater streams. Their presence can contribute to foaming, eutrophication, toxicity toward aquatic organisms, changes in pH and salinity, and reduced efficiency of wastewater treatment processes [4]. Among detergent components, phosphate-containing formulations have historically represented an important source of phosphorus in wastewater, despite legislative measures that have progressively reduced their contribution to phosphorus loads in surface waters [5]. Anionic surfactants, such as linear alkylbenzene sulphonates (LASs), are particularly relevant because they are widely used in household and cleaning products and, consequently, are among the predominant surfactants detected in wastewater streams and receiving water bodies [6].

Among the different approaches proposed for water treatment, adsorption represents one of the most widely investigated techniques, owing to its operational simplicity, relatively low cost, and effectiveness towards a broad range of pollutants [7,8]. In this context, graphene oxide (GO) has been widely investigated as a promising adsorbent material for water treatment applications, targeting different contaminants, ranging from drug molecules [9] to heavy metal ions [10].

Graphene oxide is an oxidised form of graphene, consisting of a two-dimensional layer of carbon atoms, with epoxy, hydroxyl, and carboxyl groups at the edges [11–13]. The presence of oxygen-containing functional groups gives graphene oxide a hydrophilic and chemically tunable character, enabling the formation of stable aqueous dispersions, self-assembled structures, and GO-based materials for water treatment applications [9]. The structural coexistence of sp^2 hybridised domains and oxygen-containing functional groups allows graphene oxide to engage with different targets through a variety of interaction mechanisms [14]. Thangavel and Venugopal investigated the adsorption behaviour of graphene oxide with different oxidation degrees using methylene blue as a model dye. The authors reported that adsorption performance was influenced by the oxidation degree of GO, solution pH and contact time. Higher oxidation levels resulted in increased adsorption capacity, which the authors associated with the higher amount of oxygen-containing functional groups and the increase in negative surface charge measured by zeta potential analysis [15]. Several approaches have been investigated to modify GO and improve its adsorption performance towards different pollutants. Taghiyeva et al. [16] investigated chemically modified GO for the removal of different pesticides. The modification increased the adsorption performance for several of the investigated compounds, with chlorothalonil removal increasing from 9% for pristine GO to 90% for the modified material. Other studies have explored the combination of GO with different materials or compounds. For example, GO has been combined with zeolite to improve the removal of methylene blue from water, reaching removal efficiencies of about 90% [17]. GO has also been modified with nitrogen- and sulphur-containing compounds for the removal of phenolic pollutants [18]. Similarly, aluminium-modified GO has been investigated for the removal of humic acid, achieving 91% removal compared with 81% for unmodified GO under the reported optimal conditions [19]. More recently, β -cyclodextrin has been combined with GO in a multilayer membrane for the removal of pharmaceutical and personal care products, including cimetidine, sulphamethoxazole and diclofenac [20]. These studies show how modifying or combining GO with other materials can be used to adapt its adsorption performance to different classes of pollutants.

Among the target molecules investigated for GO-based removal processes, surfactants also represent a relevant class of contaminants, because their amphiphilic structure allows

them to interact with GO through both polar functional groups and graphitic domains. Rezazadeh et al. [21] reported that graphene oxide can interact with non-ionic surfactants, particularly Triton X-100, through hydrogen bonding and π - π interactions. In a previous study, Rezazadeh et al. [22] also investigated the interaction between graphene oxide and the cationic surfactant CTAB, suggesting that the interaction is mainly driven by electrostatic forces, hydrophobic interactions, and cation- π interactions. Graphene oxide can also interact with anionic surfactants by electrostatic interaction, hydrophobic and polar interactions, as demonstrated by [23] and their study on the adsorption of laundry surfactants on graphene oxide.

To allow graphene oxide to interact efficiently with different target molecules while improving its handling, recovery, and integration into treatment systems, GO is commonly processed as freestanding membranes, incorporated into composite materials, or immobilised onto solid supports as coatings. GO membranes are widely studied for ion sieving, water-oil separation, and filtration processes, as reported in studies including water softening, dye and heavy metal removal [24]. Despite the interesting performances of GO membranes, their application for large-scale water treatment is still limited by some practical drawbacks, mainly related to pressure drops and to the difficulty of fabricating defect-free membranes on large-area substrates. For this reason, the use of porous matrices and three-dimensional cellular structures has been proposed as an alternative configuration, able to provide high surface area, good mass transfer and lower pressure drops [25,26]. In this study, the GO coating was deposited by a dip-squeeze washcoating method and used for the adsorption of $\text{Ca}^{2+}/\text{Mg}^{2+}$ ions. In a similar direction, Dotti et al. [27] prepared floating PU foams coated with rGO-TiO₂ composites, combining adsorption and photocatalytic degradation of Rhodamine B as a model dye pollutant. Immobilising GO, and, more generally, powdered active materials, onto a structured substrate can be associated with different benefits, despite the potential reduction in accessible active sites. In particular, immobilisation facilitates recovery of the adsorbent after treatment, avoiding additional solid-liquid separation steps that may be required when GO is used in dispersed form. Furthermore, dispersed GO sheets can undergo aggregation due to π - π interactions, which can reduce the accessible surface area and adsorption capacity [28]. Immobilising GO as a coating also reduces the need to handle freely dispersed GO sheets during treatment and enables the preparation of structured adsorbents that can be readily incorporated into filtration or flow-through devices. From a process perspective, conventional particulate packed beds are generally associated with higher pressure drops, whereas open-cell foams combine high porosity and interconnected channels with comparatively low resistance to flow and favourable fluid-solid contact [29]. Similar rGO-coated PU sponges have been proposed for wastewater treatment, where the polyurethane scaffold provides low density, elasticity and structural stability, while the graphene-based coating provides the functional surface [30]. Therefore, the aim of GO immobilisation in the present work is not to maximise the intrinsic accessible surface area of GO, but to balance adsorption functionality with handling, recovery and potential integration into a practical treatment device.

2. Materials and Methods

2.1. Materials

A commercial graphene oxide powder, as shown in Table 1, supplied by Graphenea Inc. (Pais Vasco, Spain), is tested to measure the adsorption properties towards a surfactant present in a standard detergent. The main composition of this standard detergent consists of standardised detergent IEC-W, bought from Ausiliari Tessili srl (Cornaredo, Italy), as represented in the next table, Table 2.

Table 1. Main physical properties of the graphene oxide powder.

Particle size D ₉₀	25–28 µm
Particle size D ₅₀	13–15 µm
Particle size D ₁₀	6–7 µm
Apparent density	0.2–0.4 g/cm ³

Table 2. IEC-W detergent composition.

Component	Specification	Tolerance ±
Linear alkyl benzene sulphonate	10%	1.0%
Ethoxylated fatty alcohol C12–14 (7 EO)	5.0%	0.5%
Fatty acid	1.0%	0.5%
Phosphonate	0.50%	0.05%
Propanediol	5.0%	0.52%
1,2-Benzisothiazolin-3-one, BIT	100 mg/kg	10 mg/kg
Sodium hydroxide	To pH 8.5	—
Demineralised water	Balance	—

The main surfactant is the linear alkylbenzene sulphonate.

2.2. Adsorption Properties

To characterise the adsorption properties, a procedure similar to previous studies [9,21,31] is adopted. In particular, a certain amount of GO powder, m_{ads} , is mixed with a certain volume, V_0 , of detergent with a concentration in water of 0.2 wt%, C_0 , with a ratio of 1 g_{GO}/kg_{det}. In a typical test, about 170 mg of GO powder was added to 170 mL of detergent solution, thermostated at the selected temperature and continuously stirred at 300 rpm. To define an adsorption curve at different times, a method proposed in different studies is used. In particular, the method consists of sampling the solution, V_{sample} , at different times to build up the adsorption curve q_t vs. time, where the adsorption capacity q_t is calculated as follows:

$$q_t = \frac{(C_0 \times V_0) - \sum_{in=1}^t (C_{in} \times V_{sample}) - (C_t \times V_{residual})}{m_{ads}} \quad (1)$$

where $V_{residual}$ is equal to the residual volume calculated as follows:

$$V_{residual} = V_0 - \sum_{i=1}^t V_{sample,i} \quad (2)$$

The equation represents three contributions. The first one, $C_0 \times V_0$, represents the initial quantity of detergent which is present at the beginning of the experiment. The term $C_t \times V_{residual}$ represents the mass of detergent present at a certain time t within the initial container batch. The term $\sum_{in=1}^t (C_{in} \times V_{sample})$ represents the cumulative amount of detergent removed from the system with the sampled aliquots.

The adsorption capacity (mg/g) was calculated as the amount of detergent adsorbed, quantified through LAS concentration, per unit mass of GO powder added to the adsorption test. Therefore, the reported adsorption capacity refers to the GO powder mass used in the experiment and not to the foam mass or to the total GO-PU composite mass.

The concentration C_t is calculated by collecting the UV spectra of the sampled aliquots after filtration by a PVDF syringe filter with an open mesh size of 0.45 µm to remove the graphene oxide powder. The UV spectra were collected using a Cary 60 UV-Vis Spectrophotometer (Agilent Technologies Italia S.p.A., Cernusco sul Naviglio, Italy) over the

200–800 nm wavelength range. A calibration curve was constructed to correlate measured absorbance with the detergent concentration, allowing the concentration of the analysed solutions to be estimated from their UV-Vis absorbance.

The adsorption tests are performed at 25 °C, 40 °C and 60 °C.

After determining the adsorption capacity of graphene oxide powder towards the target, a test to determine the capacity to remove the same chemical from a water solution by a coating of graphene oxide deposited on a foam is proposed.

2.3. Production of Graphene Oxide Coating

To deposit a coating of graphene oxide on polyurethane foams, various solutions with different concentrations are prepared. The solutions are prepared simply by dilution starting from a commercial graphene oxide solution with a concentration of 4 g/L. In particular, 2 g/L, 1 g/L, 0.5 g/L and 0.25 g/L are prepared, and their viscosity is tested. Their rheological behaviour is evaluated by measuring viscosity curves in the range from 1 to 100 s⁻¹.

The substrate consists of polyurethane open-cell foams characterised by pore densities of 20 and 60 PPI, as shown in Figure 1, and the main characteristics are indicated in Table 3.

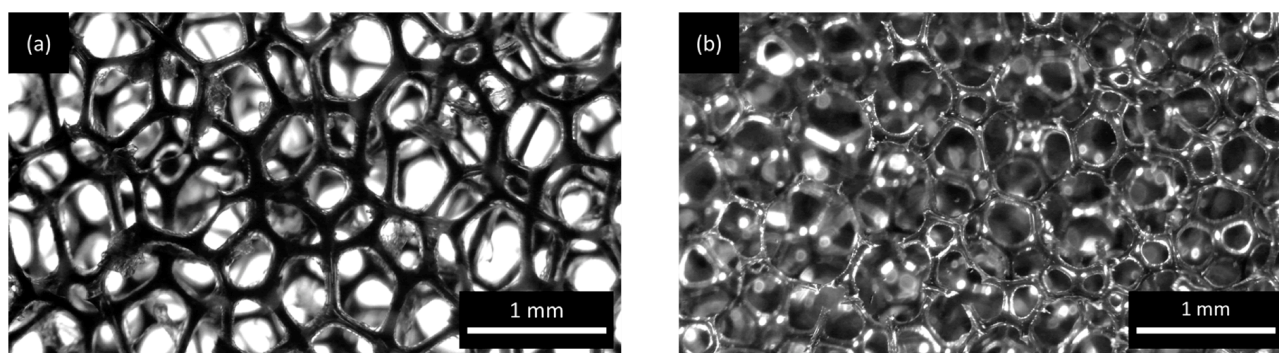


Figure 1. (a) The 20 PPI foam at 10×; (b) 60 PPI foam at 30×.

Table 3. Polyurethane foam characteristics: the strut size for 60 PPI is taken from [25], and the porosity and the geometric surface area are calculated according to [32].

	20 PPI	60 PPI
Cell diameter, mm	2.987 ± 0.123	0.674 ± 0.067
Pore size, mm	1.205 ± 0.187	0.291 ± 0.037
Density, g/cm ³	0.0347 ± 0.0011	0.0314 ± 0.0013
Strut shape	Triangle	Triangle
Strut size, mm [25]	0.335 ± 0.012	0.064 ± 0.008
Porosity, ε [32]	0.902	0.916
Geometric surface area, m ⁻¹ [32]	958	3970

The coating was produced through a soak-and-squeeze procedure. The foam, previously cut in the form of cylinders, with a diameter of 37 mm and a height of 10 mm, is soaked in the solution under test and then allowed to fully impregnate. Then it is extracted and placed between the designed equipment to squeeze the foam in a controlled way. The foams are then weighed and dried.

To characterise the shear rate characteristics of the system, a first rationalisation of the process is proposed.

In particular, a way to characterise the shear rate affecting the solution which is present in the cell of the foam is needed. To first model these two velocities of deformation applied to the foam are defined as follows in Figure 2:

$$v_c = \frac{(W - d)}{t_{nip}} \quad (3)$$

$$v_w = \frac{\varnothing_{foam}}{t_{nip}} \quad (4)$$

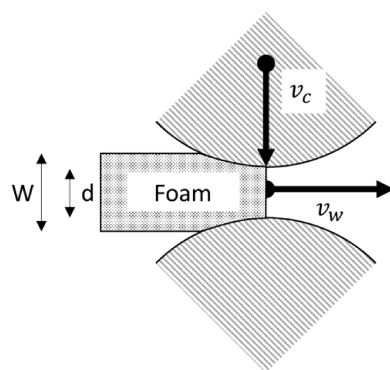


Figure 2. Schematic representation of the squeezing process.

v_c represents the velocity at which the foam is squeezed. It takes into account the initial thickness of the foam, W , the distance d between the two cylinders, and the time that is needed for the foam to pass the cylinders, t_{nip} . v_w represent the velocity of withdrawal; therefore, the time that the whole foam needs to pass the cylinders. Defining the two contributions makes it possible to firstly ideate the shear rate, $\dot{\gamma}_{eff}$, applied to the fluid within the cells, and when squeezed, tries to run out of the foam through the pores:

$$\dot{\gamma}_{eff} \propto \left(\frac{v_c}{\varnothing_{pore}} + \frac{v_w}{\varnothing_{pore}} \right) \propto \left(\frac{(W - d) + \varnothing_{foam}}{t_{nip} \cdot \varnothing_{pore}} \right) \quad (5)$$

The fixed parameters considered are listed in the next table, Table 4.

Table 4. Parameters for shear rate calculation.

Parameter	Value
W	10 mm
d	3 mm
$\varnothing_{foam}$	37 mm
t_{nip}	3 s
$\varnothing_{pores}$ 20 PPI	1.205 mm
$\varnothing_{pores}$ 60 PPI	0.291 mm

Considering the two substrates used for this study, which have different pore sizes, $\varnothing_{pore}$, it is possible to conclude that the shear rate characterising the deposition process, which is dependent on the type of foam, is 12.2 s^{-1} and 50.4 s^{-1} for 20 and 60 PPI, respectively. The higher shear rate for the 60 PPI is due to the smaller dimensions of the pores: the graphene oxide solution undergoes a higher shear rate to evacuate from the cell through the pores.

These values were then used to select the apparent viscosity representative of the deposition conditions and to interpret the coating deposition results.

The deposition of the coating is evaluated by weighing the foam before and after the deposition, after a proper drying phase, and the load is calculated as follows [25].

$$GO\ Load = \frac{m_{GO}}{m_{GO} + m_{foam}} \cdot 100 \quad (6)$$

where m_{GO} represents the mass of deposited GO and m_{foam} is the mass of the bare PU foam.

Multiple deposition cycles were performed on the foams, resulting in samples with one, two, and three consecutive coating layers.

To assess the coating distribution and the preservation of the porous structure after deposition, optical microscopy observations were performed. Images were acquired using a stereomicroscope supplied by OPTIKA S.r.l. (Ponteranica, BG, Italy), at different magnifications selected according to the foam pore size and the specific observation. In particular, 20 PPI foams were observed at $6.7\times$, whereas 60 PPI foams were observed at $15\times$. The images were used to qualitatively assess the distribution of the deposited material and possible pore blockage with increasing deposition cycles. In addition, the selected 20 PPI specimens were carefully sectioned and observed along the section to evaluate the presence of the deposited material within the internal regions of the open-cell structure.

2.4. Test on the GO-Coated Foam

The system produced as described before is tested by a filtration test. The equipment used to characterise it is shown in the next scheme, in Figure 3. In particular, the system is composed of a pump that takes the fluid, consisting of 2 L of detergent solution at a concentration of about 0.2 wt%, and passes it through a filter housing containing the coated foam, exploiting the piston flow at 2 L/min. After the housing, the fluid is collected and sampled every 200 mL, measuring the conductivity and collecting the UV spectra of the samples.

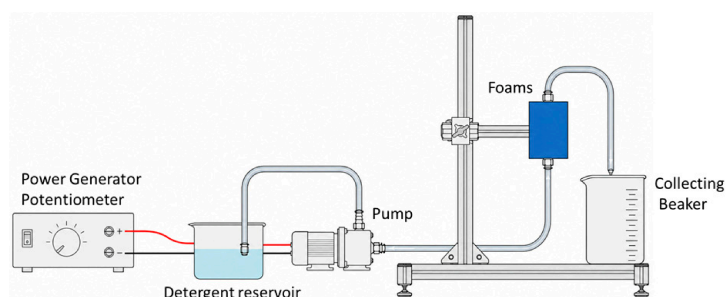


Figure 3. Setup for filtration test.

To characterise the adsorption properties of the coated foam, they are tested by letting it come into contact with a detergent solution consisting of detergent IEC-W and tap water, slightly agitated, characterised by a concentration of 0.2 wt%, for 1 h. In addition, further tests were performed to investigate the effect of selected experimental parameters on detergent concentration reduction, including the GO-to-detergent ratio, contact time, and foam configuration. For all the tests, the solutions were filtered by a syringe filter, and the UV spectra of the filtered solution were collected, following the procedure previously described for assessing the adsorption properties of the GO powder.

3. Discussion and Results

3.1. Viscosity

First of all, the viscosity curves of the different solutions are presented. The viscosity curves, including the fitting models and the associated standard deviations, are shown in the next picture, in Figure 4.

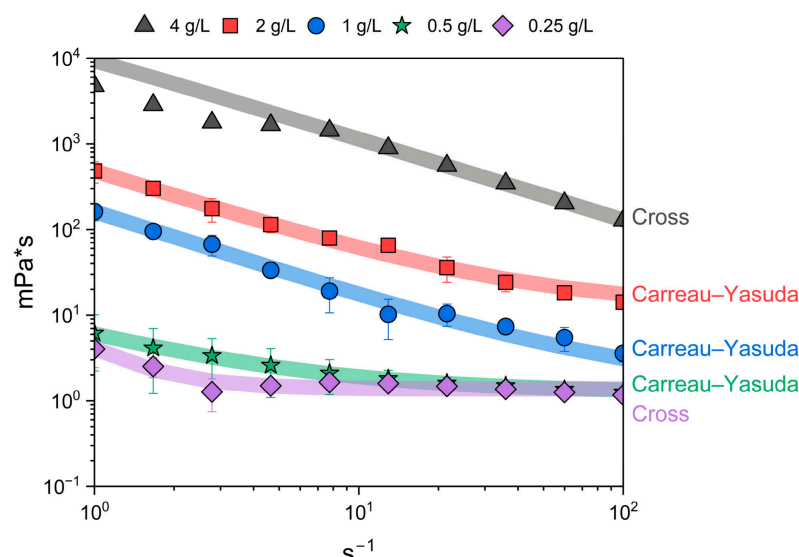


Figure 4. Viscosity curves for GO solutions; shadow lines represent fitting models and bars represent standard deviations.

Data show that the viscosity generally increases as the concentration of the GO solution increases along the tested shear rate range, due to the interaction between the graphene oxide particles above a critical volume fraction [33,34].

All the solutions present a non-Newtonian behaviour, which generally decreases from the solution characterised by a concentration of 4 g/L to that with 0.25 g/L. Fitting the data by general power laws, these results are confirmed, and the flow behaviour index, n , is reported in the next table, as shown in Table 5. Lower values of n are generally associated with more concentrated dispersions, indicating a more pronounced shear-thinning behaviour. Under the applied shear, the GO structures tend to progressively align and partially disrupt, resulting in a viscosity reduction at increasing shear rates [33].

Table 5. Flow behaviour indices for the different GO solutions.

[GO] Solution	n
4 g/L	0.056
2 g/L	0.097
1 g/L	0.028
0.5 g/L	0.573
0.25 g/L	0.713

As mentioned, the coefficient generally increases from a solution of 4 g/L to 0.25 g/L. For this reason, two models commonly used in the literature to describe shear-thinning fluids were evaluated: the Cross model and the Carreau–Yasuda model. The respective fits are reported in the previous picture as well, in Figure 4.

From the picture, Figure 4, it is possible to note that the two limit concentrations, 0.25 g/L and 4 g/L, belong to the Cross model, while the middle concentrations belong to the Carreau–Yasuda model.

For non-Newtonian GO dispersions, the apparent viscosity depends on the applied shear rate. Therefore, the apparent viscosity corresponding to the estimated shear rate for each type of foam was calculated from the fitted rheological models and used for the load analysis. The resulting values are reported in the following table, Table 6.

Table 6. Apparent viscosities of the different GO solutions calculated at 12.2 s^{-1} and 50.4 s^{-1} for the case of 20 and 60 PPI foams, respectively.

[GO] Solution	η (12.2 s^{-1}) mPa·s	η (50.4 s^{-1}) mPa·s
4 g/L	943 ± 36	246 ± 14
2 g/L	51 ± 10	19.5 ± 6.8
1 g/L	14.7 ± 3.8	4.3 ± 2.0
0.5 g/L	1.8 ± 0.6	1.4 ± 0.1
0.25 g/L	1.7 ± 0.1	1.4 ± 0.1

From the above table, it is possible to notice that the 0.25 g/L solution, at the considered shear rates, shows a behaviour very close to Newtonian. In addition, the apparent viscosity values of both the 0.25 g/L and 0.5 g/L solutions are too low to obtain a suitable deposition; therefore, these concentrations were not selected for the foam coating tests.

3.2. GO Load Assessment

The following graphs, Figures 5 and 6, show the results obtained from the coating deposition tests. In the graphs, the median GO load values are reported, while the coloured area represents the range between the first and third quartiles for each tested configuration. In particular, firstly, the GO load is reported as a function of the number of consecutive depositions, as shown in Figure 5. Each graph refers to a different GO concentration: 1 g/L, 2 g/L and 4 g/L.

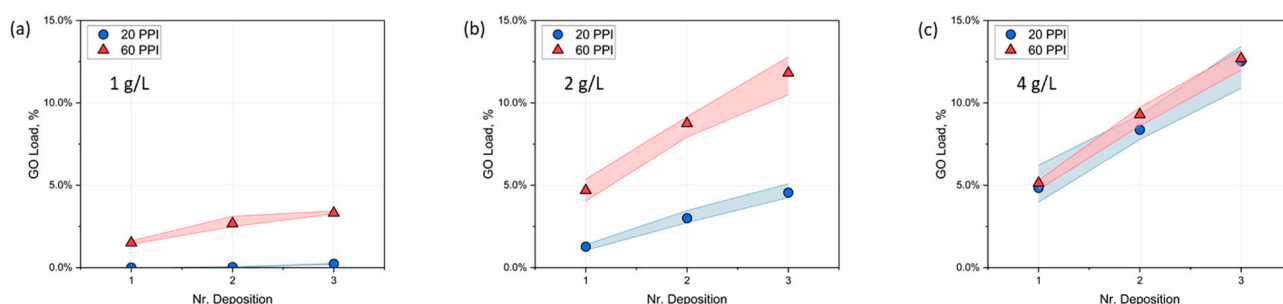


Figure 5. GO load versus number of consecutive depositions for (a) 1 g/L, (b) 2 g/L and (c) 4 g/L.

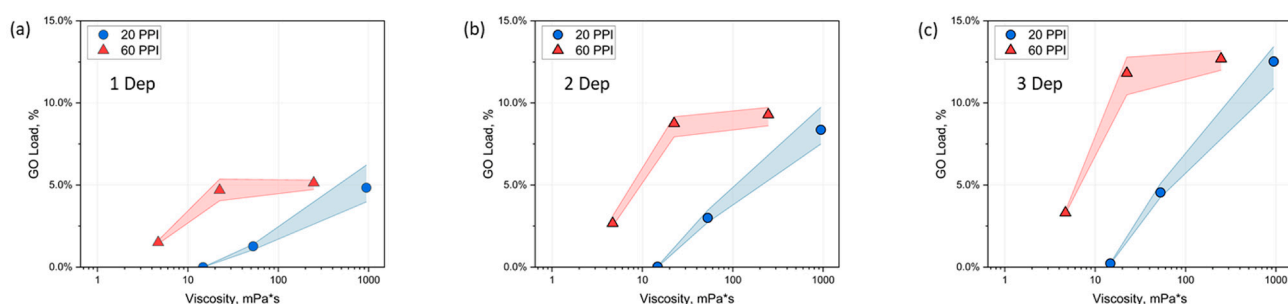


Figure 6. GO load versus apparent viscosity for (a) 1 deposition, (b) 2 depositions, and (c) 3 depositions.

From these graphs, it is possible to evaluate that, generally speaking, the GO Load increases as the number of consecutive depositions increases, for every concentration of GO evaluated. The maximum GO loads reached are about 12.7%, both for 20 and 60 PPI.

The measured GO load of 12.7 wt%, corresponding to a mass gain of approximately 14.5% relative to the pristine foam, falls within the range of coating mass gains reported for GO-containing polyurethane foam systems, which typically span approximately 8–25%

depending on the coating composition and deposition procedure [35], as depicted in the next table, Table 7.

Table 7. Literature comparison of coating mass/weight gains reported for graphene-based coatings deposited on porous polymeric supports. Different coating compositions and deposition procedures are reported.

Coating System	Depositions Cycles	Techniques	Reported Mass/Weight Gain	Reference
CS/AL/GO	10	LbL deposition	8.3%	[36]
PEI/GO + AL	12	LbL deposition	25.1%	[37]
PEI/GO + AL	12	LbL deposition	21.3%	[37]
GO	5	LbL deposition	6.6%	[38]
GO/ β -FeOOH	5	LbL deposition	12.5%	[38]
CHIT/GO	3	LbL deposition	10.2%	[39]
CHIT/GO	6	LbL deposition	13.4%	[39]
This Work	3	Soak and squeeze	14.5%	-

From Figure 5c, that represent the GO load results of the 4 g/L solution, it states that the two curves are very similar in terms of values and behaviour. This behaviour can be attributed to the higher apparent viscosity that characterises the 20 PPI foam at its characteristic shear rate, which may compensate for the more open structure, larger pores, and slightly lower porosity of the foam.

Now the GO loads with respect to the apparent viscosities of the solution for each set of consecutive depositions are reported, as shown in Figure 6.

From the three graphs, different behaviour between 20 PPI and 60 PPI for each number of consecutive depositions is observed. In particular, for the 60 PPI foam, the GO load initially increases with the apparent viscosity, but then tends to approach saturation. On the contrary, for the 20 PPI foam, the GO load continues to increase within the investigated viscosity range, without showing a clear plateau. This suggests that, for the 60 PPI foam, using a more concentrated solution is not the most effective way to further increase the deposited load. In this case, multiple depositions seem to be more useful.

After presenting the results concerning the amount of deposited graphene oxide, optical microscopy images are reported to assess the overall quality of the coating in terms of preservation of the porous structure and distribution of the deposited material within the foam.

Firstly, the effect of the number of deposition cycles on the foam structure was evaluated on 20 PPI specimens at a magnification of $6.7\times$, as shown in Figure 7. The optical images show that the cellular structure remains clearly open after one, two, and three deposition cycles, although a progressive modification of the appearance of the cell walls can be observed with increasing deposition. No evident systematic blockage of the pores is observed at the scale investigated.

A similar evaluation was performed on 60 PPI foams, as shown in Figure 8, at a magnification of $15\times$. Despite the smaller pore size, the open-cell structure remains recognisable after the investigated deposition cycles, with no evident widespread pore blockage. The images also show a progressive change in the appearance of the cell walls as the number of deposition cycles increases.

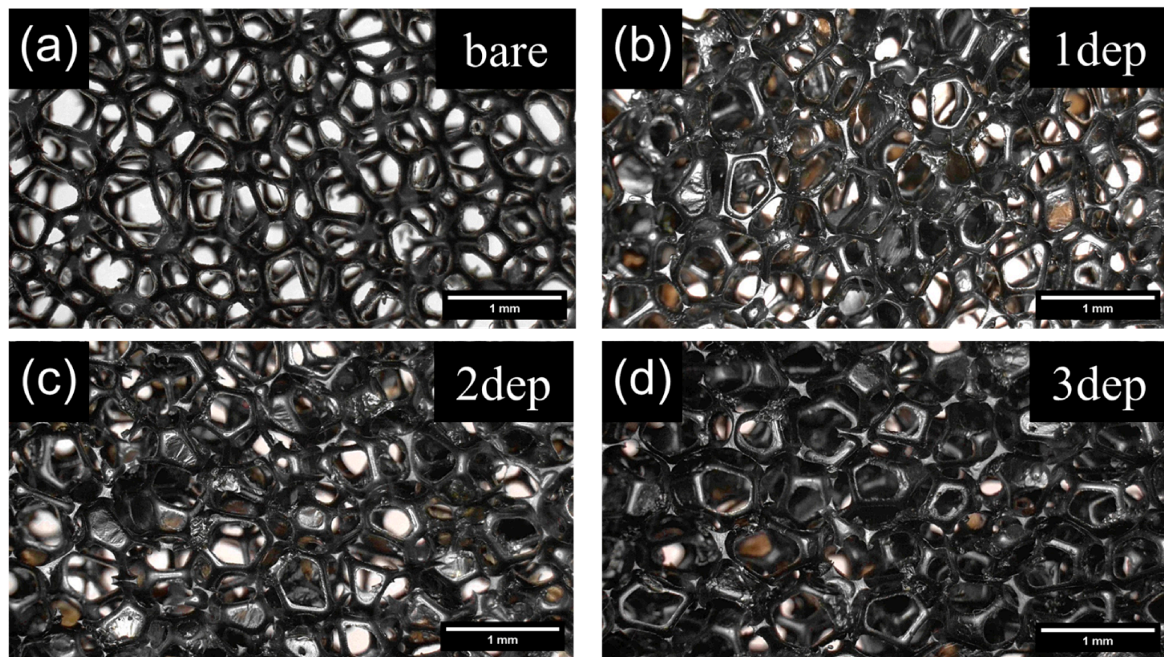


Figure 7. Representative optical microscopy images of foams at 20 PPI at magnification $6.7\times$, surface view: (a) bare, (b) 1 dep, (c) 2 dep, (d) 3 dep.

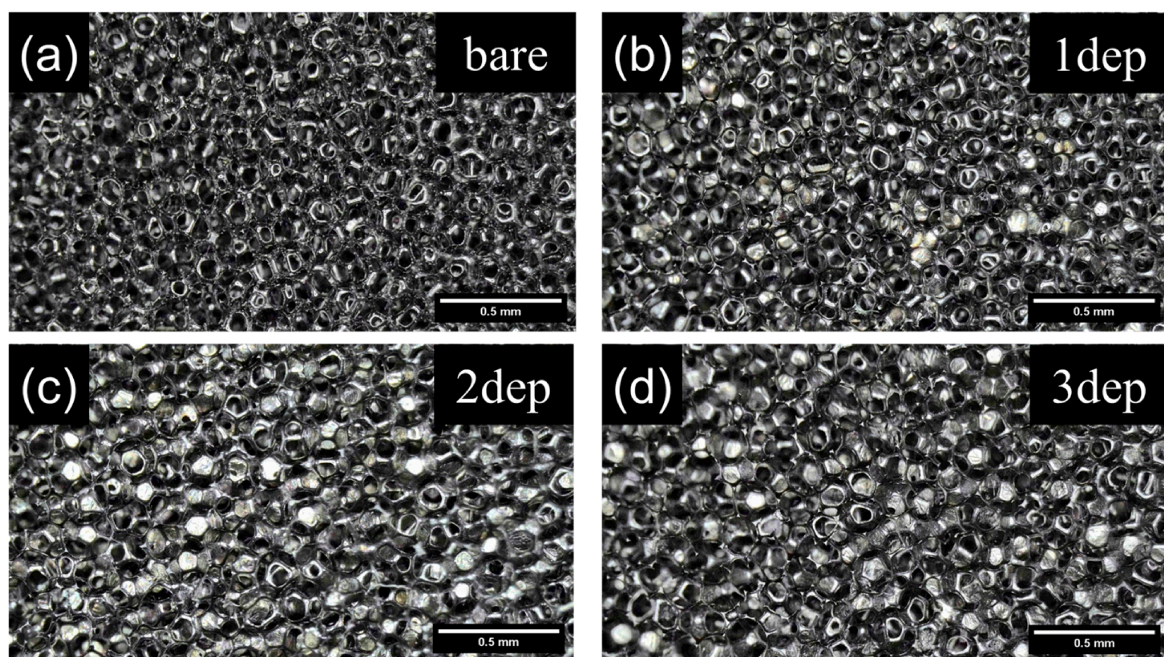


Figure 8. Representative optical microscopy images of foams at 60 PPI at magnification $15\times$, surface view: (a) bare, (b) 1 dep, (c) 2 dep, (d) 3 dep.

To further evaluate the distribution of the deposited material within the foam structure, 20 PPI white specimens were cut and observed along the thickness direction, from top (left) to bottom (right), Figure 9. The comparison between the pristine and coated specimens provides visual evidence of deposited material within the internal regions of the foam, indicating that the deposition is not restricted to the outer surfaces. This observation provides an indication of the ability of the developed deposition process to distribute the GO coating not only on the external surface but also onto the internal surfaces of the open-cell foam.

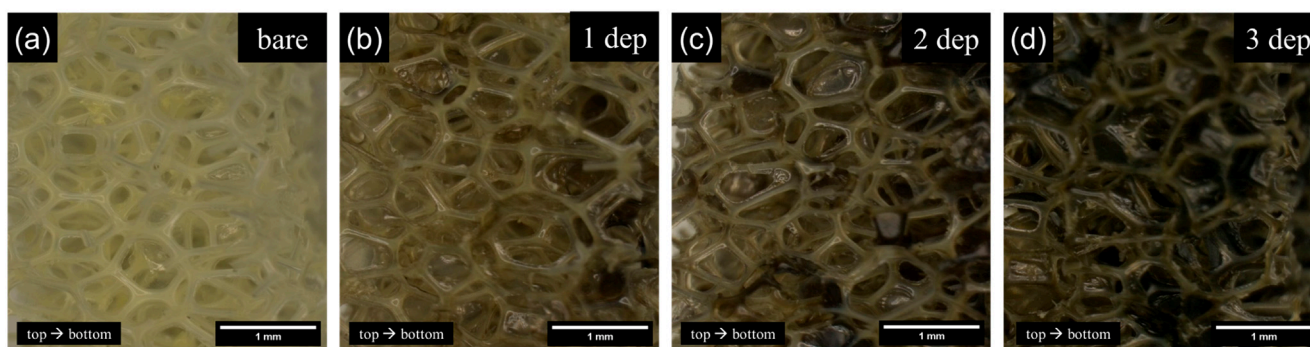


Figure 9. Representative optical microscopy images of white foams at 20 PPI at magnification $6.7\times$, cut specimens: (a) bare, (b) 1 dep, (c) 2 dep, (d) 3 dep.

3.3. Kinetic Tests

Kinetic adsorption tests were performed to investigate the adsorption properties of graphene oxide and to determine the contact time required to promote interactions between graphene oxide and the target detergent, primarily represented by linear alkylbenzene sulphonate (LAS). The UV-Vis spectrum of the detergent solution showed an absorption profile consistent with LAS, as shown in Figure 10, with the characteristic aromatic absorption band associated with the benzene ring. Since most of the other detergent components are not expected to show a strong aromatic absorption in this region, the band at 261 nm, associated with a secondary $\pi-\pi^*$ transition of the aromatic moiety [40,41], was selected as a suitable analytical wavelength to monitor the LAS-related signal within the detergent mixture. The selection of this wavelength is also supported by the previous literature on SDBS, an alkylbenzene sulphonate structurally related to LAS, in which the authors identified a well-defined absorption peak at 261 nm and used it for quantification [42]. However, due to the complex composition of the detergent formulation, this approach should not be considered as a fully selective quantification of LAS adsorption alone, but rather as an indirect evaluation of the interaction between GO and the detergent formulation as a whole. A calibration curve was then built at this wavelength and used to monitor the concentration evolution during the adsorption tests.

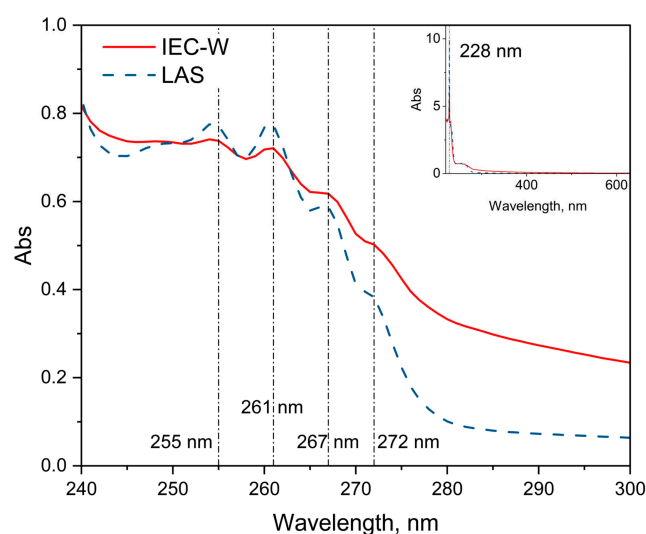


Figure 10. UV spectra of detergent IEC-W and LAS.

After gathering the spectra during the adsorption kinetics, different graphs of q_t vs. time at different temperatures were collected and presented in the next picture, as shown in Figure 11.

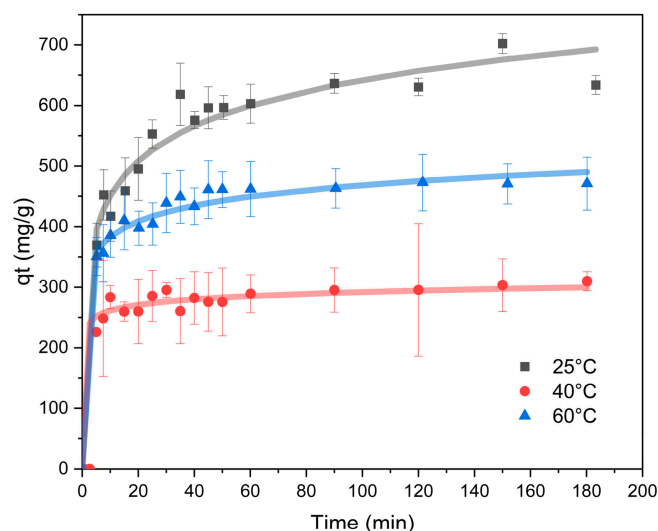


Figure 11. Detergent adsorption of detergent by graphene oxide with respect to time. The error bars represent the maximum and the minimum value of the repetitions and coloured lines represent the Elovich fitting.

Different kinetic adsorption models were considered to describe the behaviour at the three investigated temperatures: pseudo-first-order model, pseudo-second-order model and Elovich model.

The fitting performance of the three kinetic models was quantitatively compared using the coefficient of determination (R^2) and the root mean square error (RMSE) at the three investigated temperatures, as reported in Table 8.

Table 8. Fitting parameters (R^2 and RMSE) of the pseudo-first-order (PFO), pseudo-second-order (PSO), and Elovich kinetic models at 25, 40, and 60 °C.

Curve		25 °C			40 °C			60 °C		
Model		PFO	PSO	Elovich	PFO	PSO	Elovich	PFO	PSO	Elovich
R^2		0.875	0.958	0.967	0.862	0.894	0.932	0.951	0.986	0.985
RMSE		0.056	0.033	0.029	0.025	0.022	0.017	0.024	0.013	0.013

Among them, the Elovich model was able to represent the experimental data more consistently. Overall, the Elovich model provided a good description of the experimental adsorption kinetics, showing the best fit at 25 and 40 °C, while at 60 °C it still provided a very good fit comparable to that of the pseudo-second-order model.

This model is generally used for adsorption on heterogeneous surfaces, where the adsorption rate decreases with the increase in surface coverage [43,44]. In this sense, the target molecules can firstly interact with the most energetically favourable sites and then with other less favourable sites, as described by [44] to analyse Pb adsorption on activated carbon. The behaviour described by the Elovich model can be related to the structure of graphene oxide, which presents graphitic domains together with oxygen-containing functional groups, such as hydroxyl, epoxy, and carboxyl groups, providing different possible interaction sites [45].

The Elovich model, represented by the following Equation (7), is a common model that characterises carbonaceous and oxidised materials.

$$q_t = \frac{1}{\beta} \cdot \ln(1 + \alpha \cdot \beta \cdot t) \quad (7)$$

where q_t is the adsorption capacity at time t , α is the initial adsorption rate, and $1/\beta$ is related to the extent of surface coverage and the activation energy for chemisorption [44].

From the three graphs, some information can be highlighted. In particular, it is possible to first note that the initial adsorption rate is high, creating an instability in the parameter α . The terms $1/\beta$ are 83.4 mg/g, 13.2 mg/g and 37.2 mg/g for the three temperatures 25, 40 and 60 °C, respectively. This behaviour can be mainly explained by considering the complex nature of the detergent formulation. In this study, the adsorbate is a commercial detergent formulation containing several chemical components, including linear alkylbenzene sulphonate (LAS) and ethoxylated non-ionic surfactants. Therefore, the adsorption behaviour cannot be directly attributed to a single molecule but should be considered as the result of possible competitive and/or cooperative interactions among different detergent components and the GO heterogeneous surface.

Among the surfactants reported in the literature, sodium dodecylbenzene sulphonate (SDBS) represents a relevant analogue of LAS because both molecules belong to the alkylbenzene sulphonate family. Studies on SDBS adsorption onto graphene-based materials proposed that adsorption may involve a combination of hydrophobic interactions between the alkyl chain and graphene domains and π - π interactions between the aromatic ring and the sp^2 carbon structure of graphene-based materials [46]. These interactions may contribute to adsorption despite the electrostatic repulsion expected between negatively charged sulphonate groups and GO surfaces.

Hydrophobic interactions have also been identified as an important contribution for non-ionic surfactants. Studies performed with ethoxylated non-ionic surfactants on graphene-based materials suggested that the alkyl chain can interact with graphitic domains, while the ethylene oxide groups may additionally interact with oxygen-containing functionalities of graphene oxide through polar interactions and hydrogen bonding [47,48].

Since both anionic and non-ionic surfactants are simultaneously present in the investigated detergent formulation, competitive and/or cooperative adsorption phenomena may occur. In particular, surfactant mixtures can exhibit a behaviour different from the adsorption of isolated molecules because surfactant-surfactant interactions, mixed micelle formation and changes in molecular availability can affect the species available for adsorption [49,50].

Furthermore, temperature may play a role in this competitive adsorption process. Previous studies on ethoxylated non-ionic surfactants adsorbed on graphene-based materials reported a non-monotonic temperature dependence, with an increase in adsorption at intermediate temperatures [51]. This behaviour was attributed to temperature-induced changes in surfactant hydration, aggregation and interfacial interactions, which may modify the adsorption equilibrium. Such phenomena may contribute to the temperature-dependent adsorption trends observed in the present work.

In addition, temperatures above 50 °C may induce slight structural modifications of GO, such as rearrangement or clustering of oxygen-containing functional groups [52], and the observed trend is sought within temperature-dependent changes in the detergent system. To further investigate this aspect, conductivity analyses were performed at different temperatures. In particular, the molar conductivity with respect to the square root of concentration was evaluated according to the Kohlrausch approach, as shown in Figure 12, in order to investigate possible aggregation or micellisation behaviours of the detergent system. The three temperatures exhibit a very similar trend, characterised by a sharp decrease in molar conductivity at low concentrations followed by a more stable region at higher concentrations. This behaviour is commonly associated with progressive aggregation and reduction in ionic mobility in surfactant systems. The similar behaviour observed across the three investigated temperatures suggests that the detergent remains

within a comparable apparent aggregation region over the investigated temperature range. This aggregation threshold appears at concentrations higher than the initial concentration used in the kinetic test, as represented by the green dotted line in Figure 12. In particular, as mentioned before, the initial detergent concentration used during the adsorption tests was 0.2 wt%, corresponding to $0.45 \text{ (wt\%)}^{1/2}$. Considering the molar conductivity curve, the apparent aggregation concentration can be estimated at around $0.60 \text{ (wt\%)}^{1/2}$, corresponding to 0.36 wt%; therefore, the diluted detergent shows a conductivity behaviour similar to that of a weak electrolyte at concentrations close to those used in the kinetic tests.

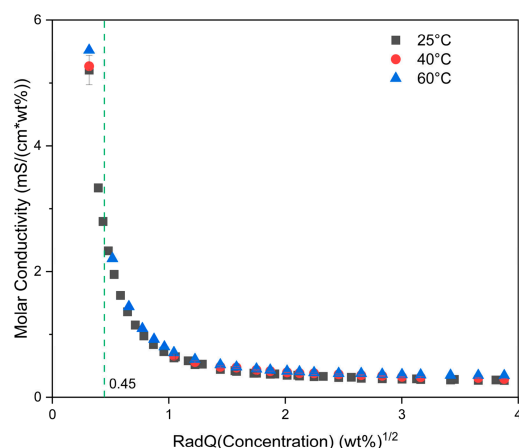


Figure 12. Molar conductivity with respect to the square root of the concentration; the green dotted line represents the initial concentration for the kinetic tests.

Considering the initial concentration used for the adsorption tests, namely 0.2 wt%, the molar conductivity curves suggest that the detergent is in a similar aggregation/micellisation region at all the investigated temperatures. Due to the complex composition of the detergent, it is not possible to define a sharp CMC value. Considering this concentration in particular, it is possible to state that the electrical conductivity increases with temperature, as shown in Figure 13. The quadratic fit and the first derivative indicate a progressive increase in the slope with temperature, without showing a clear transition point. For this reason, this behaviour can be interpreted as a gradual modification of the detergent system with temperature, rather than as a sharp transition indicating a relevant change in aggregation. This gradual and slight increase is also highlighted by the first derivative reported in Figure 13b.

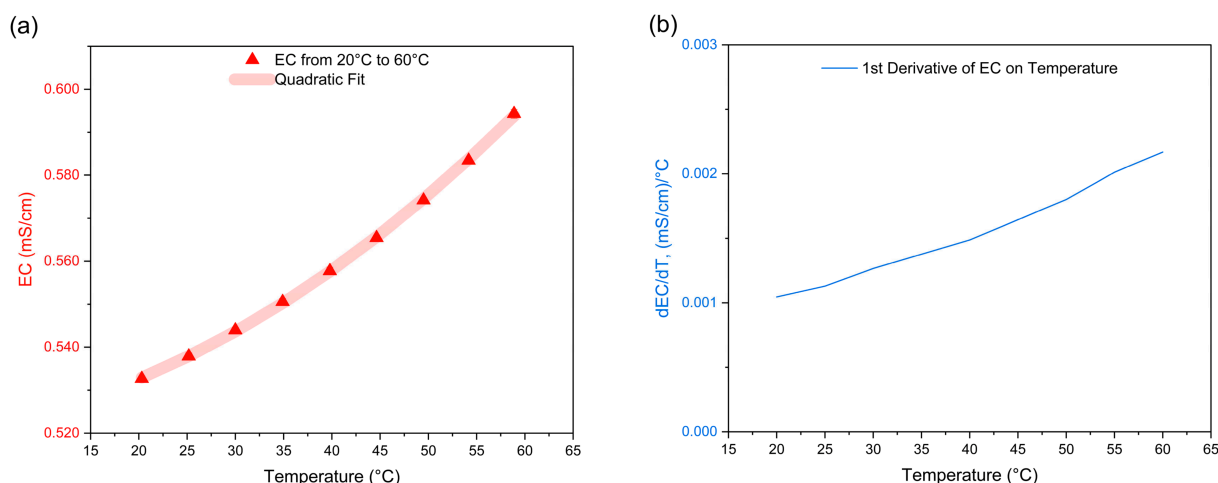


Figure 13. (a) Electrical conductivity, EC and (b) first derivative with respect to temperature at detergent concentration of 0.2 wt%.

3.4. Test on GO-Coated Foam

After studying the kinetic properties of the GO–detergent system, filtration tests were performed as described in the previous section. In particular, 2 L of water were passed through a filter containing six foams of 60 PPI, produced by three depositions of 2 g/L solution, that have a total graphene oxide amount of 0.267 g. Samples were collected every 200 mL of filtered water and analysed by both electrical conductivity and UV-Vis spectroscopy, the latter providing an estimate mainly related to the LAS adsorption. The results are reported in terms of detergent concentration as a function of the cumulative filtered volume, as shown in Figure 14. The two different approaches, represented by the two different graphs, were used to estimate the concentration of the filtered solution.

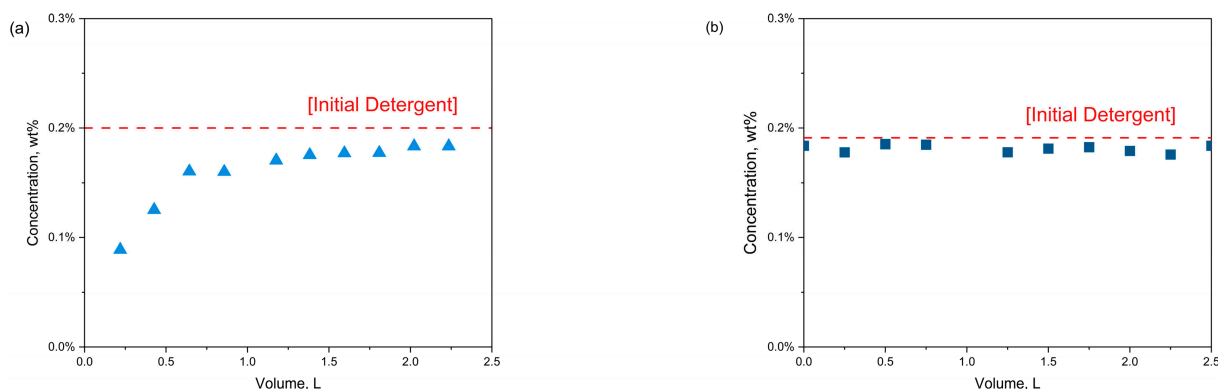


Figure 14. Concentration of filtered solution acquired by measuring conductivity (a) and collecting UV spectra (b) of the filtered solution; red dotted line represents the concentration of the initial detergent.

As shown, the conductivity measurements indicate a decrease in the estimated detergent concentration during filtration. In particular, the concentration decreases rapidly after the first 200 mL of filtered solution and then progressively increases with the cumulative filtered volume. At the end of the test, after filtering about 2 L of detergent solution, the concentration approaches the initial value, suggesting a progressive saturation of the GO-coated foam under the tested dynamic conditions. For the UV-Vis analysis, the spectra of the same sample were recorded in the 200–800 nm range, and the absorbance at 261 nm was considered for monitoring the evolution of the detergent concentration. In this case, the UV-Vis analysis does not show a clear decrease in the LAS-related signal. This could suggest that, during the filtration test, the contact time between the coated foam and the detergent solution is too short to promote a significant interaction with LAS. Therefore, the decrease observed in conductivity could be related more to other ionic species present in the detergent formulation than to a real measurable removal of LAS. From the literature, it is noted that the interaction between graphene oxide and other species, such as cations, could appear very fast [25].

Finally, after the preliminary adsorption tests, a static contact test was performed to evaluate the interaction between the GO-coated foam and the detergent solution in order to test the effectiveness of a coating of graphene oxide by increasing the contact time between the foam and the detergent. In particular, about 30 mL of 0.2 wt% detergent solution was placed in contact with a 60 PPI foam, produced by three depositions of 4 g/L GO solution, that had a GO coating of about 0.030 g, guaranteeing the ratio of 1:1 g_{GO}:kg_{det}0.2 wt% that is used during the kinetic test. The foam was immersed in the detergent solution for 1 h at 25 °C, which was selected according to the kinetic adsorption tests as a sufficient time to promote GO–detergent interactions. The decrease in concentration is reported in the next picture, including the three repetitions, as shown in Figure 15.

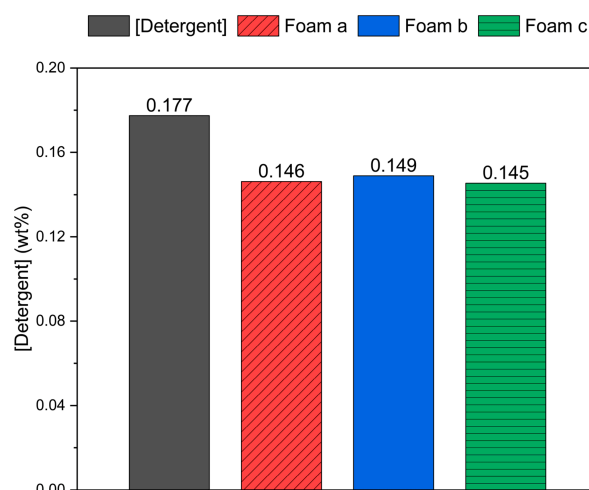


Figure 15. Adsorption test, contacting foams with detergent for 1 h at 25 °C.

As shown in Figure 15, the final detergent concentration decreased from 0.177 wt% to 0.146 wt%, 0.149 wt% and 0.146 wt% for the three repetitions, corresponding to concentration decreases of about 18%, 16% and 18%, respectively. No relevant losses of GO from the foam were observed after the test. During the kinetic test after 1 h at 25 °C, the concentration was reduced by about 37%. The lower removal compared to the kinetic test with GO powder can be due to the fact that, in the coated foam, the detergent cannot interact with all the deposited graphene oxide. In particular, the 60 PPI foam may limit the penetration of the detergent solution inside the structure, especially under slight agitation conditions. Therefore, not all the GO surface is effectively in contact with the detergent itself, reducing the final removal efficiency. This behaviour suggests that, after immobilisation, the accessibility of GO adsorption sites becomes a critical factor influencing detergent removal.

To further investigate the parameters affecting the interaction between the GO-coated foam and the detergent solution, additional adsorption tests were performed by varying the GO-to-detergent ratio, contact time and foam configuration. The complete set of results is reported in Figure 16, while the investigated conditions are summarised in Table 9.

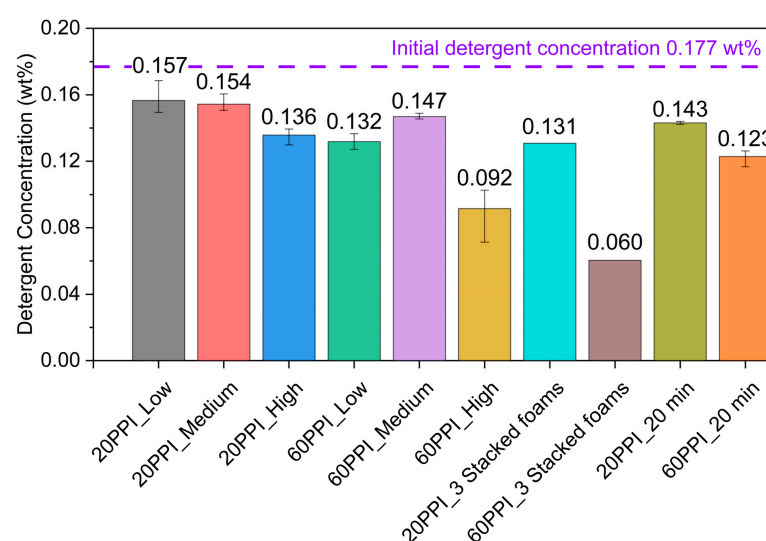


Figure 16. Summary of detergent concentration after adsorption tests performed under different experimental conditions. The dashed line represents the initial detergent concentration (0.177 wt%). Error bars indicate the range between the minimum and maximum measured values.

Table 9. Summary of the adsorption tests performed to investigate the effects of GO-to-detergent ratio, foam configuration and contact time.

Test ID	Foam Type (PPI)	Number of Stacked Foams	GO-to-Detergent Ratio (g _{GO} :kg _{det} 0.2 wt%)	Contact Time (min)
20 PPI_Low	20	1	1:3	60
20 PPI_Medium	20	1	1:1	60
20 PPI_High	20	1	3:1	60
60 PPI_Low	60	1	1:3	60
60 PPI_Medium	60	1	1:1	60
60 PPI_High	60	1	3:1	60
20 PPI_3 Stacked foams	20	3	1:1	60
60 PPI_3 Stacked foams	60	3	1:1	60
20 PPI_20 min	20	1	3:1	20
60 PPI_20 min	60	1	3:1	20

Figure 16 depicts the results obtained under the different experimental conditions investigated. The initial detergent concentration before the adsorption tests (0.177 wt%) is reported as a dashed line, while the bars represent the final detergent concentration measured after the static contact tests. The different conditions include variations in the GO-to-detergent ratio (low, medium and high), foam configuration (single and three stacked foams), and contact time, as shown in Table 9.

The results obtained for the different investigated conditions are presented below, with particular focus on the influence of GO-to-detergent ratio, contact time and foam configuration.

The first parameter investigated was the GO-to-detergent ratio. Three different conditions were evaluated, corresponding to low (1:3), medium (1:1) and high (3:1) GO availability with respect to the detergent solution.

In Figure 17, the detergent concentration reduction obtained under the different GO-to-detergent ratio conditions is reported, together with the corresponding amount of graphene oxide deposited onto the used foams. As shown in the figure, the low (1:3) and medium (1:1) GO-to-detergent ratios showed similar detergent concentration reductions for both 20 PPI and 60 PPI foams. A higher reduction was obtained when increasing the ratio to 3:1. In particular, the 20 PPI foam reached a concentration reduction of approximately 21.9%, while the 60 PPI foam reached up to approximately 47.3%.

Since the highest detergent concentration reduction was obtained at a GO-to-detergent ratio of 3:1, this condition was further investigated by reducing the contact time from 60 to 20 min. The results are reported in Figure 18.

Under the investigated conditions, the reduction in contact time did not lead to an improvement in detergent removal. On the contrary, both foam structures showed lower concentration reductions after 20 min compared with the corresponding 60 min tests. The 60 PPI foam maintained the highest performance, reaching a detergent concentration reduction of approximately 29.3%, whereas the 20 PPI foam showed a reduction of approximately 17.6%. These values are comparable with the reduction observed during the kinetic test performed with GO powder, where a reduction in detergent concentration of approximately 29% was already achieved after 20 min.

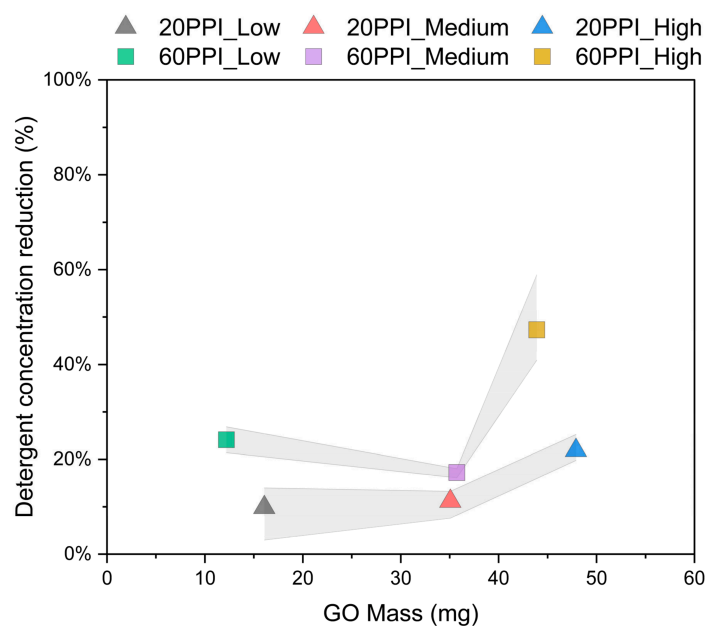


Figure 17. Effect of GO-to-detergent ratio on detergent concentration reduction; the grey areas represent the range between the maximum and the minimum values for each test.

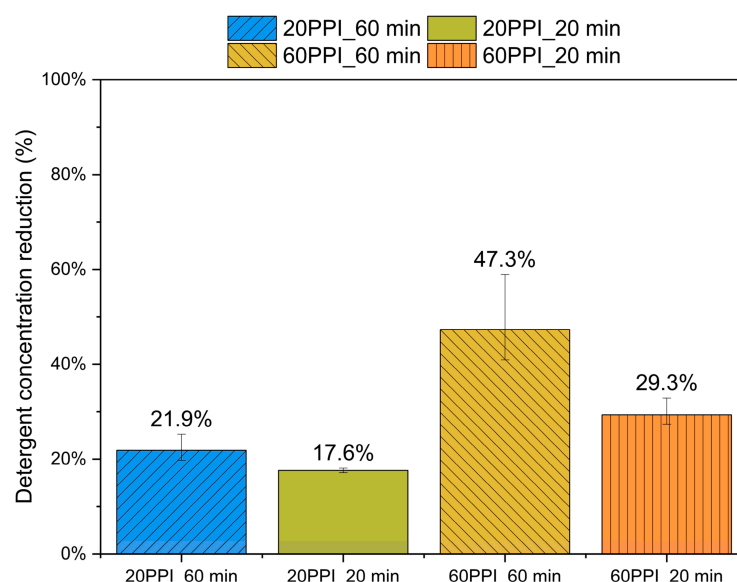


Figure 18. Effect of contact time on detergent concentration reduction at a GO-to-detergent ratio of 3:1. Error bars indicate the range between the minimum and maximum measured values.

Up to this point, the highest detergent concentration reductions were mainly obtained for the 3:1 GO-to-detergent ratio.

This behaviour can be related to the experimental conditions adopted to maintain the GO-to-detergent ratio constant. Under these conditions, the size of GO-coated foam was sufficient to occupy the liquid volume, increasing the contact area between the aqueous phase and the GO-coated surface. On the contrary, at lower GO-to-detergent ratios (1:3 and 1:1), the foam occupied only a fraction of the available liquid volume, resulting in a lower contact between the detergent solution and the immobilised GO.

To further investigate the effect of the foam-to-solution contact volume, an additional test was performed comparing three stacked foams with a single foam configuration, while maintaining the same GO-to-detergent ratio (1:1) and the same total amount of graphene oxide between the two test configurations. In this way, the three-foam configuration filled

the entire volume of the detergent solution, while keeping constant the total amount of GO and detergent solution. The results are reported in Figure 19.

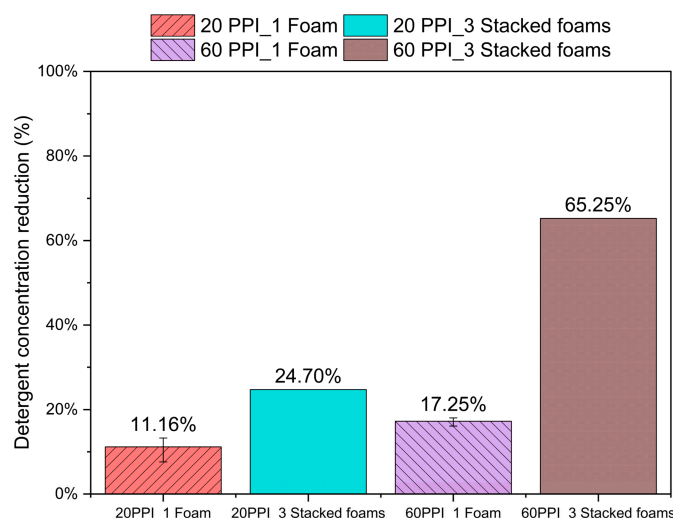


Figure 19. Effect of foam-to-solution contact volume on detergent concentration reduction at a GO-to-detergent ratio of 1:1 using single and three stacked GO-coated foams. Error bars indicate the range between the minimum and maximum measured values.

As reported in Figure 19, increasing the amount of foam within the solution resulted in a higher detergent concentration reduction for both foam structures. For the 20 PPI foam, the use of three stacked foams increased the reduction from 11.2% to 24.7%. A more pronounced improvement was observed for the 60 PPI foam, where the reduction increased from 17.3% for the single foam configuration to 65.3% when three stacked foams were used.

This behaviour suggests that, under the investigated conditions, the contact between the detergent solution and the GO-coated structure plays a relevant role in the removal process. By increasing the foam volume fraction while maintaining the same amount of graphene oxide, a larger portion of the GO-coated structure is distributed within the solution volume, potentially enhancing the interaction between detergent molecules and the immobilised GO.

4. Conclusions

This study explores a repetitive process to produce a coating of adsorbent material, in particular graphene oxide, onto a polyurethane foam and tested them for the filtration of a detergent solution. To achieve this, a process named soak and squeeze is proposed. In particular, the process consists of soaking the foam, 20 and 60 PPI, in the graphene solution at a certain concentration and the consecutive controlled squeezing of it, in order to remove the excess of the solution. Multiple depositions are, therefore, evaluated. The graphene oxide load is then calculated and evaluated with respect to the consecutive depositions and the apparent viscosity. This viscosity is calculated based on a definition of the shear characteristics of the process itself, which is defined at 12.2 s^{-1} and 50.4 s^{-1} for 20 and 60 PPI. Thanks to this process, it was possible to achieve an amount of GO load of about 12.7% for both 20 and 60 PPI. Higher GO loading values were previously obtained by our group using a similar soak-and-squeeze coating procedure [25], reaching a GO loading of approximately 27 wt%, corresponding to a mass gain of about 37% relative to the pristine foam. The difference between the two systems suggests that substrate morphology and geometrical characteristics may influence coating retention during the deposition process. A systematic investigation of the relationship between foam architecture and coating loading will be considered in future studies.

Before evaluating the filtration performance of the systems, the kinetics of GO adsorption towards the detergent are assessed at three temperatures, 25, 40 and 60 °C. The experimental data were well described by the Elovich model, indicating an adsorption behaviour typical of heterogeneous surfaces: a very fast initial uptake on the most energetically favourable active sites, followed by a progressively slower adsorption rate as the surface coverage increases. This is quite aligned with the chemical characteristics of graphene oxide. After that, the filtration performance is evaluated, first with a filtration test performed by flowing the detergent solutions through the system. It was determined that filtration, as initially considered, did not allow effective detergent removal. Although a decrease in conductivity of more than 50% was observed within the first 200 mL of filtrate, the UV-Vis spectra showed that this decrease was not associated with the removal of detergent, represented by the LAS presence. Therefore, a static contact test was proposed: by contacting the PU-coated foams with the detergent solution for 1 h, it was possible to achieve a decrease in concentration close to 20%. This reduction was subsequently improved by optimising the GO-to-detergent ratio and the foam configuration. Increasing the GO-to-detergent ratio up to 3:1 resulted in a detergent concentration reduction of approximately 47.3% for 60 PPI foams and 21.9% for 20 PPI foams. Furthermore, by increasing the foam volume fraction through the use of stacked foams, while maintaining the same GO-to-detergent ratio and total amount of graphene oxide, the reduction increased up to 65.3% for 60 PPI foams and 24.7% for 20 PPI foams. These results highlight the relevance of both the GO loading conditions and the configuration of the support in determining the removal efficiency of immobilised GO systems.

This study provides an overview of the basic methodology for the production and testing of an adsorbent coating deposited on a polymeric foam for the removal of detergents from water. While the deposition process was demonstrated to be quite reliable, to improve the filtration performance of the system, a further study of the adsorption phenomena will be necessary, because the complex nature of the standard detergent detected could produce different phenomena that interact with the material. In addition, increasing the accessibility of the surface of the foam to the detergent, using a 20 PPI instead of 60 PPI, should be beneficial.

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