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Role of magnesium-bearing silicates in the flotation of pyrite in the

presence of serpentine slimes

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Abstract: Flotation is the most effective separation solution used in sulphide ore beneficiation. In sulphide ore flotation, the interaction between the valuable sulphide minerals and the gangues are complex. Serpentine, a common magnesium-bearing silicate mineral in sulphide ores, can largely depress the flotation of the valuable sulphide minerals by adhering at their surfaces (i.e. slime-coating). In contrast, quartz can mitigate the depressing of the valuable minerals by serpentine. This work studied the effect of two common magnesium-bearing silicate minerals in sulphide ores (i.e. pyroxene and olivine) on the flotation of pyrite which was used as a model sulphide mineral. It was found that, similar to quartz, pyroxene and olivine could significantly improve the recovery of pyrite depressed by serpentine. Zeta potential measurements and turbidity experiments showed that serpentine could aggregate with pyroxene and olivine in aqueous solution via electrostatic interaction. Furthermore, DLVO calculation revealed that serpentine preferentially interacted with pyroxene and olivine rather than pyrite, resulting in increased pyrite recovery by stripping serpentine from pyrite surface.

Keyword: Flotation; Heterocoagulation; Slime coating; DLVO; Pyrite

1 Introduction

Flotation has been widely used in mineral processing for the separation and concentration of mineral ores by exploiting the differences in hydrophobicity of the various minerals in the ores. Each year, more than a billion tons of sulphide ores are beneficiated via flotation throughout the world [1-3]. Sulphide minerals are often associated with silicate gangues, including quartz, magnesium-bearing and aluminum-bearing silicates. Sulphide ore flotation is a concentration process by removing the gangue minerals from the valuable sulphide minerals. Specially, magnesium-bearing silicates need to be removed to the greatest extent as magnesium oxide (MgO) has high smelting point which can cause technical problems in the down-stream metallurgical processing [4].

Serpentine ($Mg_3Si_2O_5(OH)_4$) is a common magnesium-bearing silicate gangue in sulphide ores [5], and flotation of sulphide ores associated with serpentine is complex, owing to the interactions between sulphide minerals and serpentine, especially in the flotation of copper-nickel sulphides [6]. The easyto-slime serpentine can adhere at the sulphide mineral surfaces and restrain collector adsorption on the sulphide minerals, resulting in hydrophilic mineral surfaces [7]. The process is known as "slime coating". Slime coating is directly related to the surface charge difference between the sulphide minerals and serpentine in aqueous solution [8, 9]. Serpentine has a point of zero charge (PZC) at pH value of 9.5 while sulphide minerals normally have PZCs below 7 [10]. Flotation of sulphide minerals is normally performed under weakly alkaline conditions at which the positively charged serpentine can interact with the negatively charged sulphide minerals through electrostatic attraction [5, 11]. To overcome the adhesion of serpentine on sulphide minerals, dispersants are usually added in flotation to mitigate the slime coating [12]. Carboxy methyl cellulose (CMC) or sodium silicate can be used to disperse the ultrafine serpentine particles [9, 13, 14]. However, high reagent dosages are normally required, not only imposing detrimental impact on environment but also causing high capital cost [15].

Instead of using dispersants, it was found that the slime-coating of serpentine at sulphide mineral surface can be mitigated by the presence of quartz, a common gangue mineral in sulphide ores [5]. The PZC of quartz is about 2 in aqueous solution [16]. Quartz is much more negatively charged than sulphide minerals at weak alkaline solution. Thus, positively charged serpentine is preferred to aggregate with quartz rather than with sulphide minerals. The coagulation of particles with different characteristics (e.g. size, chemical composition, or surface charges), which irreversibly leads to a solid

or a gel-like structure in a suspension, is known as heterocoagulation [17]. The heterocoagulation between quartz and serpentine can facilitate the separation of sulphide minerals from serpentine [5].

The finding above naturally leads one to investigate the effect of other gangue minerals on the separation of sulphide minerals from serpentine. Natural serpentine is originated from the alteration of pyroxene (MgSiO₃) or olivine (Mg₂SiO₄) after hydrothermal metamorphosis [18]. Hence, serpentine usually coexists with pyroxene and olivine [19]. However, no work has been dedicated to investigate the role of pyroxene and olivine in the flotation of sulphide minerals. It is still unclear whether pyroxene and olivine act as serpentine to restrain the concentration of sulphide ores or, in contrast, act as quartz to alleviate the slime coating. Better understanding of the interactions between serpentine and other gangues could eliminate the use of dispersants in flotation of sulphide ores associated with serpentine.

In this work, a comparative study was performed to investigate the effect of quartz, pyroxene and olivine on flotation of pyrite which is used as a model material for sulphide minerals. Note that the pyrite has been coated by serpentine slimes. This allows us to study whether the presence of pyroxene and olivine can deteriorate or enhance the flotation performance of sulphide ores. In addition, pyroxene and olive are common gangue minerals not only in sulphide ores but also in some oxide minerals, such as ilmenite and chromite [20, 21]. Therefore, this research would also potentially improve the understanding of roles of these magnesium-bearing silicate gangues in the flotation of some oxide ores.

2 Experimental

2.1 Materials and reagents

Pyrite and silicate minerals (quartz, serpentine, pyroxene and olivine) (>92% pure) were obtained from different sources in China. All samples used in the experiments were crushed and ground to the particle size as desired. Pyrite sample with a particle size range of 38-150 µm was obtained through wet sieving. The silicate minerals were finely ground and the particle size distributions of them are shown in Fig. 1. Since serpentine is a group of sub-minerals, the examination by X-ray diffraction showed that it consisted of lizardite and minor chlorite. Lizardite is volumetrically the most abundant serpentine species [22].

Potassium amyl xanthate (PAX) (Tokyo Chemical Industry Co., Ltd, Japan) was used as collector and Methyl Isobutyl Carbinol (MIBC) (Sigma-Aldrich, USA) was used as frother. Sodium hydroxide (NaOH) and hydrochloric acid (HCl), for adjusting pH, were obtained from the Sinopharm Chemical Reagent Co., Ltd., China. All the reagents were of analytical grade. Deionized water was used in all experiments in this work.

2.2 Micro-flotation

Micro-flotation tests were conducted using an XFGII agitation flotation machine with a 40 mL cell at a fixed agitation speed of 1800 rpm. 50 g/L of pyrite was used in all flotation experiments in this study. 2.5 g/L of serpentine was used when needed. The mixture ratio of pyrite to serpentine was chosen based on previous studies according to industry process [5]. The other silicate mineral (pyroxene, olivine or quartz) was added at a concentration as needed. The pH was adjusted by adding HCl or NaOH stock solution. The PAX was added at a dosage as desired and 10 mg/L MIBC was used in this study. The conditioning time for PAX and MIBC was 2 min and 1 min, respectively. The concentrate was collected for 3 min in each flotation test. All flotation experiments were conducted at room temperature. The concentrates and the tailings were filtered, dried, and weighed. The concentrate and the tailings of each test were assayed to calculate the pyrite recovery.

2.3 Turbidity measurements

The mineral suspension was prepared by mixing 1 g mineral particles with 40 mL water in the flotation cell, and agitated for 3 min to disperse the solids thoroughly in water. 10 mL of the stock mineral suspension was pipetted out and diluted to 100 mL in a beaker. Then, the pH of the diluted suspension was adjusted as needed using NaOH or HCl solutions, followed by agitating at 1800 rpm for 10 min using a magnetic stirrer. After settling for 10 min, 25 mL supernatant was sampled and measured immediately using a turbidity meter (WGZ-3/3P) to determine the turbidity values.

2.4 Zeta potential measurements

Zeta potentials of pyrite and the silicate minerals were measured using a Js94H zeta potential analyzer. Mineral samples were finely ground to less than 2 μ m using an agate mortar and a pestle. For each measurement, 50 mg of mineral sample was added to 30 mL aqueous solution, magnetically stirred for

10 minutes and the pH was adjusted using HCl or NaOH solutions. All measurements were conducted in 0.1 mol/L KNO₃ solution to maintain the ionic strength.

3 Results and discussion

3.1 Effects of silicate minerals on the flotation of pyrite slime-coated by serpentine

This section presents the effect of introducing a silicate mineral on the flotation of pyrite slime-coated by serpentine at various flotation conditions by changing collector dosage, pH and the concentration of the introduced silicate mineral.

3.1.1 Collector concentration

Fig. 2a shows the effect of different silicate minerals (serpentine, pyroxene, olivine and quartz) on pyrite flotation as a function of PAX dosage at pH 9. The concentration of pyroxene, olivine and quartz was added at 2.5 g/L. The flotation of pure pyrite under different pHs is also shown in Fig. 2a for comparison. It is seen from Fig. 2a that the maximum flotation recovery of pyrite alone was achieved at the PAX dosage of 1×10^{-4} M, after which further increasing of PAX dosage did not increase the pyrite recovery. However, pyrite flotation was significantly depressed by serpentine throughout the PAX concentration varied in this study. This effect is similar to the previous study [5]. For example, the recovery of pyrite was decreased from 91% to 32% at the dosage of 1×10^{-4} M PAX. Higher collector dosage would be needed to mitigate the detrimental effect of serpentine while pyrite recovery still cannot be restored as with the absence of serpentine. For example, at the PAX dosage of 2.0×10^{-4} M, the flotation recovery of pyrite slime-coated by serpentine was increased to 54%, which is still much lower than 91%. The change is consistent with the previous studies and can be explained by slime coating of serpentine at pyrite surface [5, 12]. Differently, the pyrite recovery did not drop significantly with the presence of any of the three silicate minerals. The results indicate that the other three silicate minerals did not interact with pyrite during flotation.

Fig. 2b shows the effect of introducing pyroxene, olivine or quartz on the flotation of pyrite slimecoated by serpentine. The concentration of pyroxene, olivine and quartz was added at 7.5 g/L at a time. The flotation tests were performed at pH 9. The recovery curves of pyrite alone and pyrite slime-coated by serpentine as a function of PAX dosage are also shown in Fig. 1b for comparison. Similar to previous study showing that quartz could enhance the flotation of pentlandite and pyrite slime-coated

by serpentine [5, 15]. The present study shows that quartz could improve the flotation of pyrite slimecoated by serpentine. Hence, it is confirmed that quartz could mitigate the detrimental effect of serpentine on sulphide mineral flotation. In addition, it is observed that the presence of pyroxene and olivine can also enhance the flotation of pyrite slime-coated by serpentine throughout the PAX concentration varied in this study. For example, the recovery of pyrite slime-coated by serpentine was improved from 32% to 58% for olivine and 70% for pyroxene, respectively, at the dosage of 1×10^{-4} M PAX. It can be found in Fig. 2b that pyroxene exhibits the strongest ability to alleviate the detrimental effect of serpentine on the flotation of pyrite, followed by quartz, and olivine shows the relatively weakest effect.

3.1.2 pH

Fig. 3a shows the effect of silicate minerals on the pyrite flotation recovery at different pHs with PAX dosage of 1×10^{-4} M. The solids concentration of pyroxene, olivine and quartz was added at 2.5 g/L. It is shown that the recovery of pyrite alone increased with increasing pH and achieved the maximum recovery of 91% at pH 9, after which the recovery decreased with increasing pH while pyrite recovery in the presence of serpentine was sharply decreased with increasing pH throughout the entire range. For instance, the recovery of pyrite with the presence of serpentine was decreased to 32% at pH 9. Similar to Fig. 2a, no significant effect of the other three silicate minerals (pyroxene, olivine and quartz) on pyrite flotation is observed within the pH range varied in this study.

Fig. 3b shows that the addition of pyroxene, olivine or quartz can significantly improve the recovery of pyrite which has been slime-coated by serpentine. The concentration of pyroxene, olivine and quartz was added at 7.5 g/L. The flotation tests were performed with 1×10^4 M of PAX. The flotation recovery of pyrite alone and pyrite slime-coated by serpentine as a function of PAX dosage is also shown in Fig. 2b for comparison. The results in Fig. 3b show that the recovery of pyrite slime-coated by serpentine can be improved at a wide pH range between 4 and 12 when pyroxene, olivine or quartz is introduced. It is interesting to note that the peak recovery of pyrite is shifted from weak alkali pH to acid pH when a silicate mineral is introduced, revealing that the interactions between pyrite and silicate minerals are highly pH dependent. In addition, similar to the order observed in Fig. 2b, pyroxene exhibited the strongest ability to alleviate the detrimental effect of serpentine on the flotation of pyrite, followed by quartz and olivine showed the relatively weakest effect.

3.1.3 Concentration of the introduced silicate minerals

Fig. 4 shows the effect of the concentration of the introduced silicate minerals on the flotation of pyrite slime-coated by serpentine. The flotation tests were performed at pH 9 with 1×10^{-4} M of PAX. As shown in Fig. 4, the effects of the three introduced silicate minerals on pyrite flotation follow the same trend. The recovery of pyrite slime-coated by serpentine increased significantly with increasing the concentration of each introduced silicate mineral up to 7.5 g/L, after which the flotation recovery of pyrite only increased slightly over increasing the concentration of an introduced silicate mineral at 7.5 to 15.0 g/L before decreasing at higher concentration. The excessive amount of silicate minerals may lead to the adverse consequence that residual silicate minerals require the consumption of collector after sufficiently interact with serpentine. Thus, the concentration of the silicate mineral introduced should be properly determined.

3.2 Heterocoagulation between the introduced silicate minerals and serpentine

The results above show that introducing a silicate mineral (pyroxene, quartz or olivine) significantly improves the flotation recovery of pyrite slime-coated by serpentine. It is speculated that the interactions between the serpentine and the introduced minerals occurred via heterocoagulation. Electrostatic interaction is expected to be the factor that dominates the process of heterocoagulation as it is strongly affected by pH as shown in Fig. 3b.

Fig. 5 shows the measured zeta potential profiles of the silicate minerals and pyrite as a function of pH in aqueous solution. The measured point of zero charge (PZC) of pyrite is at the pH value of 6.5, which is consistent with literature [3, 23, 24]. The PZC of serpentine is at the pH value of 10.3, which is also consistent with the previous study [5]. As shown in Fig. 4, the PZCs of pyroxene, quartz and olivine are at the pH values of 3.2, 2.1 and 2.9, respectively, which are also consistent with the results reported in literatures [25-28]. Flotation of sulphide ores is normally performed at neutral or weak alkaline conditions [3]. Under such pH condition, serpentine is positively charged, while pyrite, pyroxene, quartz and olivine are all negatively charged. Since the PZCs of the three introduced silicate minerals (i.e. pyroxene, quartz and olivine) are lower than that of pyrite, the three silicate minerals are more negatively charged than pyrite, serpentine would preferentially interact with the three silicate particles rather than pyrite. The process is defined as heterocoagulaiton via electrostatic attraction. This may

explain why the presence of the three silicate minerals can improve the flotation recovery of pyrite with the presence of serpentine.

Turbidity measurements was performed to investigate the heterocoagulation interaction between the introduced silicate minerals and serpentine. Turbidity value varies largely between different minerals as it is affected by the size and shape of the particles [29]. To compare the turbidity changes caused by the heterocoagulation between serpentine and the different silicate minerals, the degree of heterocoagulation (*DH*) is defined in this study as:

$$DH = \frac{\overline{\tau} - \tau_{\rm M}}{\overline{\tau}} \times 100\% \tag{1}$$

where $\tau_{\rm M}$ is the suspension turbidity of silicate-serpentine mixture while $\bar{\tau}$ is the average of turbidity values of the single silicate mineral and serpentine. If there is an heterocoagulation interaction between the silicate mineral and serpentine, the value of $\tau_{\rm M}$ will be significantly less than that of $\bar{\tau}$. The heterocoagulation between serpentine and each of the three introduced silicate minerals was studied individually. The results are summarized in Table 1. Note that the mixture of serpentine and each silicate mineral is at the mass ratio of 1:1.

On the basis of Eq. (1) and Table 1, the *DH* between serpentine and each silicate mineral was calculated and the results are shown in Fig. 6. It shows that the *DH* of the three mixtures (pyroxene-serpentine, quartz-serpentine and olivine-serpentine) is pH dependent and follows the same trend. The *DH* values increased with increasing pH from 2.5 and reached the maximum of 22.2% for pyroxene, 20.2% for quartz and 16.1% for olivine at pH 9, before decreasing at higher pHs. In addition, the DH follows the order of pyroxene \approx quartz > olivine, which is consistent with their effect on improving the recovery of pyrite slime-coated by serpentine (see Section 3.1).

3.3 DLVO calculation for the interactions between silicate minerals and pyrite

To further understand the competitive interaction between serpentine and the other minerals, the interaction energies between serpentine and the other minerals were calculated through application of DLVO theory, which allows the quantitative prediction of the interaction between colloidal particles in aqueous solution. The total interaction energy $V_{\rm T}$, ignoring non-DLVO terms is [30]:

$$V_{\rm T} = V_{\rm E} + V_{\rm W} \tag{2}$$

where $V_{\rm w}$ is the Van der Waals energy and $V_{\rm E}$ is the electrical energy. The electrical interaction energy versus separation distance for two spherical particles carrying electrical double layers can be written as [31]:

$$V_{\rm E} = \frac{\pi \varepsilon_0 \varepsilon_{\rm r} R_1 R_2}{(R_1 + R_2)} \left\{ 2\psi_1 \psi_2 \ln \left[\frac{1 + \exp(-\kappa H)}{1 - \exp(-\kappa H)} \right] + \left(\psi_1^2 + \psi_2^2\right) \ln \left[1 - \exp(-2\kappa H) \right] \right\}$$
(3)

where ε_0 is the permittivity in vacuum, ε_r is the relative dielectric permittivity of the solvent. R₁ and R₂ refer to the radii of two approaching mineral particles which are obtained as D50 from Fig. 1. ψ_1 , ψ_2 are the surface potentials of the minerals which can be substituted by the measured zeta potential values shown in Fig. 5 when contact time between the particles is short [32]. In the present calculation, the interactions between minerals were performed at pHs 3, 5, 9 and 11. H represents the distance between particles. κ^{-1} is the DEBYE length which is the thickness of the double layer and is given by [33]:

$$\kappa^{-1} = \frac{0.304}{\sqrt{C_{\text{NaCl}}}} \tag{4}$$

where C_{NaCl} represents the concentration of NaCl with unit mol/L. In the present study, κ^{-1} equal to 30.4 nm for 0.1 mM NaCl.

The van der Waals interaction energy is [28]:

$$V_{\rm w} = -\frac{A_{132}}{6H} \left(\frac{R_1 R_2}{R_1 + R_2}\right) \tag{5}$$

where A_{132} refers to the Hamaker constant, the Hamaker constants for the interaction between serpentine and other minerals in aqueous solution are not available in literature, but can be estimated as [34]:

$$A_{123} \approx (\sqrt{A_{11}} - \sqrt{A_{33}})(\sqrt{A_{22}} - \sqrt{A_{33}})$$
(6)

where A_{11} , A_{22} and A_{33} are the Hamaker constants of minerals 1, 2 and water 3 in vacuum. According to the references, the Hamaker constants of minerals and water in a vacuum are listed as 12.9×10^{-20} J for pyrite [15] and 6.3×10^{-20} J for quartz [35]. On the other hand, the Hamaker constant of 6.28×10^{-20} J for serpentine, 8.6×10^{-20} J for pyroxene, 7.4×10^{-20} J for olivine and 3.7×10^{-20} J for water is given by [36]:

$$A_{i} = \frac{3}{4}k_{B}T\left(\frac{\varepsilon_{i}-1}{\varepsilon_{i}+1}\right)^{2} + \frac{3hV_{a}}{16\sqrt{2}}\frac{(n_{i}^{2}-1)^{2}}{(n_{i}^{2}+1)^{1.5}}$$
(7)

where ε_i is the static dielectric constant, n_i is the refractive index, h is the Planck's constant and V_a is the main absorption frequency in the UV region. The data for each material is given in Table 2 [26, 37-40].

Fig. 7 shows the total interaction energy $V_{\rm T}$ between serpentine and the other silicate minerals based on the equations 3-8 at different pHs. At pH 3, the total inter-particle force between pyrite and serpentine is repulsive whereas it is attractive between serpentine and the other minerals (i.e. olivine, quartz and pyroxene). As increasing pH to 5, the interaction energy between pyrite and serpentine became attractive, at which serpentine could coat the pyrite surface so as to depress its floatability. Meanwhile, the attractive energies between the other silicate minerals and serpentine are enlarged. The attractive energies between serpentine and the other minerals follow the order of quartz~ pyroxene> olivine >pyrite. Thus, serpentine particles tend to interact with silicate minerals than pyrite. When increasing pH to 9, the order remains unchanged but the attractive energies reach their peaks. At higher pH of 11, however, the energies between serpentine and the other minerals became repulsive. Fig. 7 explains why the heterocoagulation between serpentine and the introduced silicate minerals is pH dependent (Fig. 6) and the recovery of pyrite depressed by serpentine is also pH dependent with the presence of silicate minerals (Fig. 3b). Note that the calculated attractive energies of serpentinequartz and pyroxene-serpentine are close while pyroxene exhibited slightly better impact than quartz on alleviating the detrimental effect of serpentine on the flotation of pyrite as shown in Fig. 3b. It is probably resulted from the fact that the DLVO calculation was conducted using the average particle size of each mineral, which may not fully represent the real particle size distribution, and might have caused slight deviation on determining the interaction between serpentine and each silicate mineral.

Fig. 8 shows a schematic model to illustrate the effects of silicate minerals on the flotation of pyrite particles coated by serpentine at pH 9 where the maximum degree of heterocogualtion between serpentine and the introduced silicate minerals was achieved. As shown in Fig. 8a, the slime coating of hydrophilic serpentine at pyrite surface is resulted from the electrostatic attraction between the negatively charged pyrite and the positively charged serpentine. In contrast, Fig. 8b shows the change of surface characteristics of serpentine after the addition of a silicate mineral. The introduced silicate minerals with negative charges are repulsive to pyrite which also carries the negative charges. In the meantime, due to the stronger attraction energy between the silicate mineral and serpentine, a large proportion of serpentine slimes was stripped from the pyrite surface and coagulated with the silicate mineral.

4 Conclusions

Sulphide minerals can be significantly depressed by Mg-bearing serpentine in flotation. This work focused on the effect of other two common Mg-bearing gangue minerals in sulphide ores (i.e. pyroxene and olivine) on the flotation of sulphide minerals. Pyrite was used as a model sulphide mineral in this study. It was found that pyroxene and olivine had no detrimental effect on pyrite flotation. In contrast, similar to quartz, they could significantly improve the recovery of pyrite depressed by serpentine. The comparative study showed that their effect on improving the recovery of pyrite followed the order of pyroxene \approx quartz> olivine.

Heterocoagulation between serpentine and pyroxene and olivine was observed via turbidity measurement and was pH dependent. The zeta potential measurements implied that the heterocoagulation between serpentine and pyroxene and olivine was governed by electrostatic interaction. In addition, DLVO calculation revealed that serpentine preferentially interacted with pyroxene and olivine rather than pyrite. At alkaline pH where sulphide ore flotation is routinely conducted, serpentine surface is positively charged while pyrite, pyroxene and olivine are negatively charged. Since pyrite has higher PZC than that of pyroxene and olivine, mitigating the depressing of pyrite by serpentine in flotation.

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	Turbidity of Single Mineral (NTU)				Turbidity of Mixture (NTU)		
рН	Serpentine	Pyroxene	Quartz	Olivine	Serpentine+ Pyroxene	Serpentine+ Quartz	Serpentine+ Olivine
2.5	665	743	410	924	690	521	782
4.5	566	828	411	1021	648	450	761
6.0	558	826	468	1052	612	460	748
7.5	565	914	473	1120	626	448	768
9.0	521	916	487	1174	559	402	711
10.0	464	923	524	1184	575	412	711
11.5	470	905	418	1192	663	430	816

Table 1. Turbidity of silicate-serpentine mixtures and single minerals at different pHs.

Samples	Dielectric constant (\mathcal{E}_i)	Refractive index (n_i)	References
Serpentine	11.48	1.55	[37, 38]
Pyroxene	6.15	1.65	[39]
Olivine	6.77	1.67	[26, 37]
Water	78.50	1.33	[40]

Table 2. The values used in the calculation of Hamaker constant.

Highlights

- Pyroxene and olivine have no detrimental effect on pyrite flotation;
- The presence of pyroxene or olivine improves the flotation recovery of pyrite depressed by serpentine;
- Serpentine preferentially interacts with pyroxene and olivine rather than pyrite;
- Heterocoagulation between serpentine and pyroxene as well as olivine is governed by electrostatic interaction.



Graphics Abstract



Figure 1





Figure 3



Figure 4



Figure 5



Figure 6



