Water Science & Technology



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Water Science & Technology Vol 87 No 5, 1072 doi: 10.2166/wst.2023.040

Microplastics and organics – A comparative study of sorption of triclosan and malachite green onto polyethylene

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ABSTRACT

This study aims to elucidate interaction of organics with microplastics in a comparative manner via the use of two model compounds (i.e., triclosan (TCS) and malachite green (MG)) having different physicochemical properties, onto polyethylene (PE). TCS, is hydrophobic with low solubility, while MG is hydrophilic with high aqueous solubility. Kinetic studies indicate faster sorption ($t_{eq} = 24$ h) and equilibrium studies show much higher capacity ($q_e = 6,921 \mu g/g$) for TCS, when compared to those of MG ($t_{eq} = 5$ d, $q_e = 221 \mu g/g$). While pseudo-kinetic model fits sorption of both organics to PE, equilibrium isotherms as well as the results on effect of particle size and pH indicate dissimilar sorption mechanisms. Considering pH_{PZC} = 2, observation of favourable sorption of TCS in acidic regions and sorption being unaffected by particle size was explained by TCS sorption to be dominated by hydrophobic interactions in amorph regions of PE. Higher removal of MG was observed at lower surface charge of PE, and a clear favourable impact of surface area on MG sorptive capacity pointed to the presence of non-specific van der Waals type interactions on the surface of PE. Mechanistic evaluations presented here contribute to our understanding of interaction of MPs with organics in aquatic ecosystems.

Key words: hydrophobic interaction, malachite green, mechanism, microplastics, sorption, triclosan

HIGHLIGHTS

- Lower solubility clearly results in higher sorption affinity for organics onto PE.
- Sorption kinetics are rapid for TCS, while slow for MG.
- pH and particle size of PE affect sorption of TCS and MG differently.
- Hydrophobic interactions are dominant for TCS sorption on PE.
- Non-specific interactions on PE surface are suggested for MG sorption on PE.

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1. INTRODUCTION

Plastic pollution is now discussed on the scale of being a planetary boundary threat, owing to exposure, fate, and ecotoxicological hazards related to weathering plastics (Arp *et al.* 2021). The total amount of plastics produced had reached 830 million tons by 2015 (Dai *et al.* 2022). More than 150 million tonnes of plastic waste are estimated to have accumulated in the oceans since 1980 (European Commission 2018). Plastic particles with a size less than 5 mm, i.e., microplastics (MPs), raise a special concern due to their potential to act as vectors in the transport of organic compounds (OCs) to biota (Rochman *et al.* 2013). Even though it is still a matter of debate whether MP-sorbed OCs can cause further ecological risk to organisms through MP ingestion, it is certain that they create concern due to constituting a potential entry route of trace OCs into the food web. Considering the wide range of MP types/sizes and OCs that exist, it is critical to examine interactions between MPs and OCs.

Ubiquitous nature of MPs and potential for their interaction with a wide array of OCs in various ecosystems necessitates detailed examination of the interactions among them. Indeed, interaction of OCs with MPs are scrutinized by numerous recent review studies (Mei *et al.* 2020; Wang *et al.* 2020; Fu *et al.* 2021; Costigan *et al.* 2022). Main mechanisms are put forth as: hydrophobic interactions, partitioning (into amorph regions of the MPs), electrostatic interactions, and interactions on MP surfaces/crystalline regions (e.g., H-bonding, π - π interactions, halogen bonding), with multiple mechanisms generally acting together. For OCs and non-aromatic MPs such as polyethylene (PE), Mei *et al.* (2020) citing Hüffer & Hofmann (2016) primarily suggest non-specific van der Waals interactions. For example, Li *et al.* (2018) reported that there is a significant relationship between log K_{ow} values and sorption capacities of investigated antibiotics species on the five different MPs tested. Indeed, Costigan *et al.* (2022) underline hydrophobicity of the OC (i.e., log K_{ow}) to be a good surrogate for sorptive capacity of some polymer types and OCs, though reporting hydrophobicity alone may not suffice. Sorption behaviour of OCs on MPs depend on the structure and properties of both the OCs and the MPs of concern.

Two OCs are used as model compounds in this study for a comparative evaluation of their sorption on medium-to-highdensity PE. Triclosan (TCS) is an antimicrobial agent, common bactericide, and disinfectant, confirmed to be a persistent endocrine disruptor as well as bio-accumulative for aquatic and other organisms in the environment (Ma *et al.* 2019; Verdú *et al.* 2021). Currently, restrictions are placed by the EU for TCS use (European Commission 2018). Malachite green (MG) is a synthetic organic compound, and it is used as a fungicide, industrial dye, food colouring agent, food additive, and medical disinfectant (Lin *et al.* 2020). This organic dye is cytotoxic to mammalian cells and causes liver and kidney disease (Culp *et al.* 2006).

The aim of this study is to elucidate sorption mechanisms influential during interaction of organic compounds with PE by the use of two model compounds. TCS and MG were selected owing to their dissimilar physicochemical properties; while TCS has low aqueous solubility (0.01 g/L) and high hydrophobicity (log $K_{ow} = 4.78$), MG has high aqueous solubility (40 g/L) and low hydrophobicity (log $K_{ow} = 0.62$). Insight into sorption mechanisms would enable broader conclusions to be made for numerous OCs coexisting with microplastics in aquatic environments.

2. MATERIALS AND METHODS

2.1. Materials

Polyethylene was selected in this study because it is the most common type of plastic used and wasted (Xia *et al.* 2021). PE was purchased from a local water tank manufacturer in powder form. Prior to use, MPs were sieved, washed in an ultrasonic bath for 30 min, and dried at 30 °C (Lin *et al.* 2020). Triclosan (Ehrenstorfer) and Malachite Green (Isolab) were purchased in solid form, having higher than 99% purity.

2.2. Characterization of microplastics

MPs were characterized via diamond crystal ATR-FTIR analysis carried out at METU Central Laboratory (MERLAB) using IFS/66S, Hyperion 1000. Crystallinity and melting temperature were found by Perkin Elmer Diamond Differential Scanning Calorimetry (DSC), multi-point N₂ BET specific surface area (for 24 h degassing at 50 °C) and zeta potential and the point of zero charge (pH_{PZC}) for the microplastic sample were analysed at MERLAB using MALVERN Nano ZS90. ASTM (2017) method was followed in our laboratories to determine particle size distribution of mixed MPs.

2.3. Analysis of triclosan and malachite green

Stock solutions of TCS were prepared by dissolving it in ethanol in amber vials and stored at 4 °C in the dark (Verdú *et al.* 2021). Working solutions were prepared immediately prior to experiments. MG solutions at desired concentrations were prepared from a stock, which was prepared at least 2 h prior to use, and continuously stirred until use for complete dissolution of MG crystals. TCS and MG were measured directly using a UV/Vis Spectrophotometer (HACH DR6000) at the wavelength of their maximum absorbance, i.e., 279 and 617 nm, respectively. USEPA (2016) method was followed for determination of the method detection limit (MDL) and method quantitation limit (MQL). MG and TCS solutions were confirmed to be stable within the duration of experiments (OECD 2000).

2.4. Sorption experiments

OECD method (2000) was mainly followed during sorption experiments and shortly described here. Batch sorption experiments were carried out in triplicates using 40 mL amber glass vials with Teflon-lined caps. Prepared vials were shaken horizontally at 200 rpm and 25 ± 2 °C in an incubating shaker (N-Biyotek NB-205 VL, S.Korea). Experiments were conducted for determination of equilibration time, isotherms, effect of particle size (d_p), and pH. Except for experiments conducted to observe the effect of particle size, 250–500 µm size PE was used in all experiments. The highest percentage of MPs (40%) found in the environment belong to this size range (Costigan *et al.* 2022). Unless otherwise stated, S/L was 10 and 25 g/L for TCS and MG, respectively. Due to sorption on glassware, all results are presented as control (i.e., vials without MPs) corrected. Unless specified, TCS sorption experiments were conducted at pH 6, considering its pKa of 8.1. MG has two pKa values (i.e., 6.9 and 10.3), experiments were performed at unadjusted pH (approximately 8.5).

Kinetic experiments were conducted at 8 ppm for TCS, and 10 and 40 ppm for MG. At the predetermined times (e.g., 0, 6, 20, 24, 48, and 72 h), the liquid portion was separated from PE. All isotherm experiments were performed at t_{eq} which is 24 h for TCS and 5 days for MG. Adsorption isotherm experiments were performed for TCS and PE by changing the PE amount in the sorption vials (i.e., at solid to liquid ratio, S/L of 0.5, 1, 2, 5, 10, 15, 20, 25, 30, 40 g/L) while for MG by changing the initial concentration (i.e., 0.1, 1, 5, 8, 10, 25, 40 ppm). In particle size experiments, TCS and MG sorption were tested with PE of size ranges 106–180, 250–500, 850–1,000 μ m. Equilibrium sorption experiments for MG were carried out using mixed size particles as well. Particle size experiments were carried out with 10 ppm MG at S/L of 25 g/L and 8 ppm TCS at S/L of 5 g/L. To investigate the effect of pH on sorption, experiments were performed at three different pH values, 6, unadjusted (approximately 8)

and 10 for TCS. These values are selected by keeping in mind the acidity constant of TCS, i.e., 8.1. For MG the experiments were performed at pH 4 and unadjusted (approximately 8.5). During pH adjustment, H_2SO_4 and NaOH were used. Error bars in figures indicate standard deviation of triplicates.

3. RESULTS AND DISCUSSION

3.1. Characterization of microplastics

Main characteristics of the microplastic used can be seen in Table 1. FTIR analysis confirmed material to be polyethylene. The density (0.935 g/cm³) falls within typical densities observed in the literature, e.g., 0.93 g/cm³ by Hüffer & Hofmann (2016) reported for medium-density PE, or 0.92 and 0.94 g/cm³ for LDPE and HDPE, respectively, reported by Puckowski *et al.* (2021). Accordingly, MP sample was identified as medium-to-high-density polyethylene. Moreover, the particle size distribution analysis showed 77.4% of the particles to be in the range of 250–500 μ m. Lastly, according to the zeta potential and point of zero charge (pH_{PZC}) analysis, the surface charge of PE was confirmed to be negative at any point beyond pH 2.

3.2. Sorption kinetics

Kinetic sorption experiments indicated sorption of TCS onto PE was very fast, with no significant change in TCS concentration or sorption capacity after 8 h (Figure 1(a)). On the other hand, sorption kinetics of MG was quite different, depicting oscillating removal efficiencies after a few days even after repetitive testing, at both high (40 ppm) and low (10 ppm) initial concentration (Figure 1(b)). Affinity of TCS on PE was higher than that of MG, as can be seen from the rapid sorption kinetics and removal efficiencies for TCS (78.6%) and MG (68.9% for 10 ppm). Time to reach equilibrium (t_{eq}) was decided as 24 h and 5 days, for TCS and MG, respectively.

Kinetic data were fitted to pseudo-first-order (PFO) and pseudo-second-order (PSO) kinetic models to evaluate sorption mechanisms. The linearized form of equations are as follows:

Pseudo first-order kinetic model (Espinoza et al. 2019):

 $\ln\left(q_{\rm e}-q_{\rm t}\right)=\ln(q_{\rm e})-k_1t$

Pseudo-second-order kinetic model (Espinoza et al. 2019):

$$\frac{t}{q_{\rm t}} = \frac{1}{k_2 q_{\rm e}^2} + \frac{t}{q_{\rm e}}$$

The results (Table 2) show that PSO much better fits the data when compared to PFO for both TCS ($R^2 = 0.98$) and MG ($R^2 = 0.91$). However, for both chemicals, the sorption capacities obtained from models are different from the experimental sorption capacity results, indicating overlapping sorption mechanisms. Sorption of MG on PE (Zhong *et al.* 2022) and nylon (Lin *et al.* 2020) type microplastics were both satisfactorily fitted to the PSO model. Furthermore, Chen *et al.* (2021) and Ma *et al.* (2019) state that high agreement with PSO model indicates chemical adsorption to be the rate-limiting step for sorption of TCS on PE (Chen *et al.* 2021) and PVC (Ma *et al.* 2019). Lastly, similar to the present study, Chen *et al.* (2021) also observed fast sorption kinetics for TCS with PE as the sorbent, when compared to the polystyrene or soil they tested.

Table 1	Main	characteristics	of t	he microp	lastic	used
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Туре	Medium-to-high-density PE
Density	0.935 g/cm^3
Melting temperature	126 °C
Crystallinity (DSC)	49.4%
BET specific surface area	$0.1034 \text{ m}^2/\text{g} \; (< 1.5 \text{ m}^2/\text{g})$
Surface charge (mV)	2.52 (pH = 2) -29.8 (pH = 4) -25.0 (pH = 6) -184 (pH = 8)



Figure 1 | Sorption kinetics of (a) TCS and (b) MG onto polyethylene.

Table 2 | Pseudo first-order and pseudo-second-order kinetic model parameters for TCS and MG (10 ppm) sorption on PE

Kinetic model	Parameter	TCS	MG
Pseudo-first-order	$k_1~(\mathrm{h}^{-1})$	-0.0196	-0.0508
	$q_{\rm e}$ (µg/g)	30.4	69.8
	R^2	0.01	0.07
Pseudo-second-order	$k_2((\mu g/g \cdot h)^{-1})$	-0.0009	-0.0169
	$q_{\rm e}$ (µg/g)	476.2	153.9
	R^2	0.98	0.91

TCS sorption kinetic studies were also conducted with smaller (106–180 µm) and larger (850–1,000 µm) particle sizes of PE, yielding the same t_{eq} of 24 h, and conformity to PSO model ($R^2 > 0.98$ for both d_p , results not shown). An initial concentration (i.e., 8 ppm) close to aqueous solubility (i.e., 10 ppm) of TCS was used in the studies, which may partly be the reason for the same t_{eq} to be applicable at various particle sizes. Also, as can be seen from Figure 1(a), sorption of TCS onto PE reaches completion well before 24 h, so this t_{eq} is actually conservative and may take into account slightly different times for different particle sizes, all reaching equilibrium before 24 h.

3.3. Sorption isotherm

Sorption isotherms for TCS and MG onto PE show very different behaviour from each other (Figure 2). While TCS shows an almost linear sorption onto PE (Figure 2(a)), MG shows first an increasing and then a decreasing trend, with maximum sorption obtained around 8 ppm (Figure 2(b)). Endo & Koelmans (2016) report sorption onto rubbery polymers to be generally linear. A linear sorption is also associated with partitioning as the dominant mechanism, meaning partitioning into the amorphous regions of the polymer, such as those present in medium-to-high-density polyethylene (Endo & Koelmans 2016), used in this study. Also, Tong *et al.* (2020) report that adsorption of a hydrophobic compound such as TCS shows linear sorption onto PE and polyhdroxybutyrate. Furthermore, Yu & Bi (2016) state that hydrophobic partitioning of undissociated phenolic species into organic matter is linear. TCS is a neutral hydrophobic compound in its undissociated form, since experiments were conducted at a pH well below its pKa. Considering the pH_{pzc} of PE to be 2, rather than an electrostatic attraction-based sorption, hydrophobic sorption mechanism would be expected. The maximum adsorption capacity is measured as $6,921 \mu g/g$ for TCS.

On the other hand, for MG, the maximum adsorption capacity is measured as $221.3 \,\mu$ g/g and this capacity decreases in concentrations lower and higher than 8 ppm (Figure 2(b)). Adsorption study of MG on PE and PVC by Zhong *et al.* (2022) and on modified PET performed by Ünlü *et al.* (2020) shows that as the initial concentration of MG increases, the



Figure 2 | Sorption isotherms of (a) TCS and (b) MG onto polyethylene.

adsorption capacity increases, with capacities reported as 4,520 μ g/g for PE (Zhong *et al.* 2022) and approximately 2,500 μ g/g for modified PET fibres (Ünlü *et al.* 2020). Also, the results of Lin *et al.* (2020) which has a similar initial MG concentration with this study showed that nylon has a much higher adsorption capacity (approximately 5,400 μ g/g) than PE. Overall, MG shows a nonlinear trend and a much lower adsorption affinity towards the PE used in this study when compared to PE and other plastic types reported in the literature. From the perspective of the sorbent, Endo & Koelmans (2016) state that sorption of organic chemicals to glassy polymers (i.e., high crystallinity) follows nonlinear isotherms. In our case, we observed that for the same polymer having medium crystallinity (i.e., 49.4%), both trends could be observed, depending on the organic compound.

3.4. The effect of particle size

Equilibrium sorption experiments were carried out to understand the effect of three different particle size ranges on sorption capacity. The size ranges selected for this study, i.e., 106-180, 250-500, and 850-1,000 µm do not constitute uniformity within size ranges, yet, the aim was to compare sorption of model compounds for the most representative particle size group, i.e., 250–500 µm, and one group from lower and one group from higher particle size range. Nevertheless, as emphasized by Costigan et al. (2022), the specific surface areas for MPs used in sorption studies in the literature are typically less than $5 \text{ m}^2/\text{g}$, which constitutes relatively low surface areas. It was observed in the present study that regardless of the distribution uniformity of the particle size ranges, for the hydrophobic compound, TCS, there was no consistent trend for change in sorption with increasing or decreasing particle size. A similar observation was made by Bhagat et al. (2022), with phenanthrene onto HDPE where particle size did not have a major effect on sorption affinity. Mostly, studies indicate an increase in sorption capacity with decreasing particle size, owing to increased specific surface area (SSA), such as Li et al. (2019). In their study, SSA of polystyrene for the particle size closest to our 250–500 μ m was 0.58 m²/g (215 μ m) and 1.88 m²/g (107 μ m) while ours was measured to be less than 0.10 m²/g. However, Li et al. (2019) also report no major difference between triclosan sorption on the two particle sizes that have the lowest SSA (i.e., 0.67 m²/g for 151 μ m and 0.58 m²/g for 215 μ m). Hence, change in sorption capacity may not be as easily observed owing possibly to the relatively low change in an already low surface area. Submicron size nanoplastic particles would be expected to have a much higher surface area, but nano size range PE was out of the scope of the current study.

No net trend in sorption with changing particle size could be observed for TCS (Figure 3(a)), while a clear inverse relationship of increasing removal efficiency with decreasing particle size was apparent for MG onto PE (Figure 3(b)). According to the results, it can be concluded that particle size did not have a major effect on sorption capacity of TCS onto PE, which is already much higher than that of MG. On the other hand, the maximum sorption capacity for MG (Figure 3(b)) was obtained with the lowest particle size at 348.35 μ /g. Sorption onto 250–500 μ m and mixed size PE are observed to be very close to



Figure 3 | The sorption capacity of (a) TCS and (b) MG on PE having various particle sizes.

each other. This can be attributed to the MP size distribution since 77.4% of PE used in this study is in the size range of $250-500 \,\mu$ m.

Dissimilar responses of TCS and MG to particle size of PE also point to a clear difference in sorption mechanisms by which each organic compound sorbs onto PE. If indeed TCS partitions into the amorph regions of PE, it would be expected to be less influenced by change of particle size or surface area. While any electrostatic or surface interaction of the more hydrophilic organic, MG with PE (e.g., van der Waals) could be expected to change with a change in surface area.

3.5. The effect of pH

Both TCS and MG are ionizable, therefore pH is expected to have an impact on sorption of these compounds onto PE. According to the results, for TCS, when pH increases, sorption capacity of PE for TCS decreases (Figure 4(a)). As Ma *et al.* (2019) stated, when the pH > pKa, TCS will be in the form of TCS⁻ as a result of dissociation of its proton. On the contrary, when pH < pKa, TCS is in its undissociated form. Ma *et al.* (2019) discuss that the dissociation of TCS has an impact on



Figure 4 | Effect of pH on sorption of (a) TCS and (b) MG on polyethylene type microplastic.

adsorption by increasing the repulsion between TCS⁻ and their PVC type microplastic. Since the pH_{PZC} of our PE is 2, beyond which the surface is negatively charged, we expect an electrostatic repulsion between TCS⁻ and PE at pH 10, which is in line with the experimental findings. In addition, at pH 10, removal of TCS is 51%, yet a significant sorption of TCS⁻ also occurs on glass walls as indicated by reduction of TCS concentration in the control vials (i.e., 35%). When pH decreases, on the other hand, TCS removal increases to 86.4%, with no more than 6% decrease observed in control vials.

On the other hand, pH affects sorption of MG onto PE differently (Figure 4(b)). At pH 4, we expect MG to be in its dicationic form (since pH < pKa1), while at pH 8.5, in its cationic form (since pKa1 < pH < pKa2). Dicationic MG sorbs much less on PE and on glass walls (as indicated by MG-only results in Figure 4(b)). Cationic MG, on the other hand, shows a higher affinity for PE at pH 8.5, but there is also appreciable interaction of MG with glass walls (Figure 4(b)). Even though MG is highly soluble, we still see notable sorption affinity towards PE at pH 8.5. Dicationic MG has much less affinity for PE, which may indicate that the sorption of MG onto PE may predominantly take place from the single bonded neutral nitrogen side of the molecule, or through non-specific van der Waals interactions at the PE surface. Zhong *et al.* (2022) also observed an increase in MG sorption with increasing pH. However, their mechanistic explanation of the sorption phenomena is quite different. The pH_{PZC} of their PE sample is 4.3 and they explained the increasing sorption capacity with dominance of electrostatic attraction between cationic MG and increasing negative charge on PE surface beyond pH 4.3. In our case, after the pH_{PZC} of 2, the most negative surface charge is measured at pH 4 (-30 mV) and a slightly lower one (-20 mV) at pH 8. If electrostatic interactions had dominated, we would have observed higher sorption of the dicationic MG onto negatively charged PE at pH 4, which is not the case.

3.6. Comparison of sorption mechanisms

TCS and MG displaying dissimilar responses to particle size and pH can be explained by their varying interactions with PE. TCS portrays its highest affinity for PE in its neutral form, whereas MG is sorbed more favourably in its cationic form, as opposed to its dicationic form. Although surface interaction may not be excluded, we believe it does not constitute a major mechanism for the case of TCS. Particle size experiments indicate sorption of MG taking place on the surface of PE possibly through non-specific van der Waals type interactions, while partitioning into amorph regions of the polymer seems valid for TCS. The non-polar and non-aromatic PE structure would not enable H-bonding or halogen bonding with TCS or MG. Vastly different solubilities were observed to correspond to very different sorption capabilities, such that PE had 30 times the sorption capacity for the less soluble TCS, when compared to the 4000 times more soluble MG. Our results suggest hydrophobic interactions to be present for both model compounds.

4. CONCLUSIONS

Sorption of two physiochemically different model organics, namely, triclosan, a hydrophobic compound, and malachite green, a hydrophilic dye, is investigated for their sorption behaviour onto medium-to-high-density polyethylene type microplastics. Results indicated very different behaviour for two compounds, such that sorption was very fast for TCS, while slow for MG, even though kinetic sorption data for both followed pseudo-second-order kinetic model. Isotherm studies showed a mainly linear increasing trend for TCS, while it was nonlinear and decreasing for MG. Observations from the effect of particle size of PE and pH of solution suggested TCS sorption to be dominated by partitioning, or hydrophobic interactions in amorph regions of PE, while sorption of MG did not follow the possibly expected electrostatic interaction, but indicated more towards non-specific van der Waals type interactions on the surface of PE. This study indicates that complex interactions dominate sorption of organics with microplastics and comparative evaluation of literature may not always yield similar results even for the same compound and polymer type. Studies such as the present one, providing detailed microplastic characterization and experimental procedures, are expected to provide valuable input to contribute to shedding light on the complex interactions between two ubiquitous components of aquatic ecosystems: microplastics and organics.

AUTHOR CONTRIBUTIONS

Gökçe Çiftçi validated the article, investigated the project, analysed and interpreted the data, and wrote the original draft. Ülkü Dide Türkeli validated the article, investigated the project, analysed and interpreted the data, and wrote the original draft. Elif Yaren Özen validated the article, investigated the project, analysed and interpreted the data, and wrote the original draft. Melek Özdemir investigated the project and wrote the original draft. Faika Dilek Sanin conceptualized the whole article and wrote the review and edited the article. İpek İmamoğlu conceptualized the whole article, supervised the work, wrote the review and edited the article, visualized the article, conducted funding acquisition, and administered the project.

FUNDING

This study was funded by the Scientific and Technological Research Council of Turkey Grant No: 220N044.

DATA AVAILABILITY STATEMENT

All relevant data are included in the paper or its Supplementary Information.

CONFLICT OF INTEREST

The authors declare there is no conflict.

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First received 21 September 2022; accepted in revised form 27 January 2023. Available online 9 February 2023