REACH RESTRICTION SUPPORT – LEAD IN FISHING TACKLE AND AMMUNITION (PART 7)

FINAL REPORT TO ECHA FROM WCA

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EXECUTIVE SUMMARY

In this brief review we have investigated recent evidence, claiming that the use of steel shot at shooting ranges where lead shot is still present will accelerate the weathering of both types of shot. In turn this will lead to increased lead or iron release that will pose potential environmental risks to ground and surface waters.

The key evidence supporting this hypothesis has been generated from laboratory synthetic solution experiments conducted in the absence of soil, thus limiting environmental relevance. The evidence of increased shot weathering, when both lead and steel shot are co-occurring, is also somewhat confounded by the lack of suitable controls in the testing solutions. Field-based evidence does not support the suggested claims regarding accelerated lead migration or iron impacts upon surface and ground waters.

Chemical speciation modelling indicates that additions of iron into the soil porewater will reduce lead availability due to the formation of precipitates that lead binds to, except under acidic conditions where iron does not precipitate readily. The binding of lead species to organic matter or iron hydroxide precipitates reduces the potential for lead to be mobilised or cause toxicity. Overall, the addition of iron to soils from the use of steel shot is likely to reduce, rather than increase, the availability and mobility of lead already present in the soil.

The outcome of this review of recent data suggests no changes are required in the conclusions given from ECHA’s previous assessments.

CONTENTS

1 INTRODUCTION

This brief assessment is to provide a technical review of recent open-source evidence on the potential environmental risks posed from the use of steel shot at shooting ranges previously used with lead shot.

1.1 Aims

The aims of this report are to provide ECHA with an assessment of stakeholder comments related to the claim that use of steel gunshot on shooting ranges, as an alternative to lead, will mobilise lead and other metals. Specific points addressed are:

- The scientific/technical robustness of the major claims regarding the impact of steel gunshot on the mobility of lead and other metals in soils at shooting ranges and determination of whether the conclusions of a previous assessment on this topic need to be revised;
- The likelihood of increased lead release from shot over soils in the presence of steel shot, including undertaking of simple solubility modelling to confirm the technical feasibility of such elevated lead release and likely conditions under which it may occur;
- Critical assessment of the evidence given in the publications provided during the stakeholder consultation.

1.2 Report structure

After this brief introduction, Section 2 of this report provides an assessment of the recent open-source evidence produced by Lisin et al. (2022) and reviews the technical feasibility of the conclusions drawn by Lisin et al. using solution speciation modelling of soil solutions under a range of pH, dissolved organic carbon, iron and lead chemistries. In Section 3 we provide brief conclusions from this evidence review.

2 INFORMATION REVIEW

Assessment of Lisin et al. 2022.

An assessment has been undertaken of the scientific/technical robustness of the major claims regarding the impact of steel gunshot on the mobility of lead in soils at shooting ranges as given by Lisin et al. (2022), a study finalised after a preliminary description of the study was submitted by FITASC/ISSF in the stakeholder consultation on the Annex XV report[1]. Specifically, the paper outlines experimentation in which lead shot and steel shot are exposed, separately and together, to three synthetic solutions for 100 days. The solutions were:

- Rainwater, pH 6.1 and EC of 27 µS cm^{-1} – termed by the authors as atmospheric precipitation;
- The same rainfall, but with additions of nitric and sulphuric acid, to reduce the pH to 4, EC to 30 µS cm^{-1} – called acid precipitation; and finally,
- A solution, with addition of ammonium acetate to give pH 4.8 and EC of 4500 µS cm^{-1} – the authors suggest this is a representation of soil solution, specifically of a wetland environment.

The results are used to infer the likely outcomes associated with the use of steel shot on shooting ranges at which lead shot was previously used. The key conclusions the study draws are (as given by the authors in the paper, below, edited for brevity):

- Steel shot is weathered more rapidly than lead shot, especially in the presence of lead, forming flocs in solution; mobile soluble iron compounds and complexes may result in uncontrollable growth of the pollution of environment;
- The primary hazard is the potential increase in soil pollution (since the possible increase in the depth of lead penetration) and groundwater by mobile lead species because the presence of steel shot increases lead shot transformation by 4–8 times and promotes its decapsulation (in the absence of steel shot, lead is low active and accumulates in the upper centimetres of the soil profile);
- Where there is a high level of lead recycling (up to 90%), the primary environmental hazard is the iron pollution of underground water due to the fact that the presence of lead increases steel shot transformation by 1.3–1.7 times;
- Steel shot and its corrosion products in the form of iron hydroxides are transported from shooting areas into water bodies and watercourses by surface runoff.
- Infiltration of soluble iron compounds in the soil may lead to an increased iron content in the groundwater (water from natural sources consumed without any treatment).

These conclusions, and the data on which they have been based are assessed below.

The paper is titled an ‘environmental study’, assessing transformation of shot under …’environmental factors’. The experimentation described by the authors unfortunately does

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[1] https://www.fitasc.com/upload/images/echa_mai_2021/20210504_addendum_may21_to_fitasc_contribution_july20.pdf

lack some environmental relevance[2], for several key reasons, many of which were previously identified in ECHA 2020 (specifically in reference to the study by Hurley 2004), but include:

- Critically, there is an absence of soil in the test system. Figure 2.1 illustrates the importance of soils in influencing both the sorption behaviour and solution speciation of metals. Soils are physically, chemically, and biologically active and can buffer imposed change. As the receiving medium for the steel and lead shot it is imperative that soils are included in any assessment of behaviour and fate of a trace element if it is to be considered to have environmental relevance.
- The solutions used in the experimentation are synthetic, i.e. distilled waters amended to meet pH and electrical conductivity under hypothetical conditions. They are considered by the authors to be representative of normal rainwater, acid rain and a wetland environmental. The rainwater is pH 6.1, the acid rain pH 4.0 and pH 4.8 for the wetlands. These latter two preparations are somewhat environmentally extreme, especially the wetland solution. While this latter scenario is considered to be out of scope by ECHA for this work (and the restriction proposal with which it is concerned), the solution nevertheless has an electrical conductivity far in excess of those likely to be found in natural wetlands (e.g. Gerla et al. 2013) and the use of ammonium acetate is a very poor representation of natural organic matter in terms of complexation behaviour. It is not clear from the paper what the oxygen status is of these experimental solutions, which will be of potential importance where the focus is upon weathering and oxidation processes.
- It has long been established that solid:solution experimentation focussed on solute chemistry tends to be more reflective of environmental conditions when the solid to solution ratio is relatively high i.e. in soils the solid content would be higher than the solution under most circumstances, e.g. solid:solution ratio could be 1:10, but preferably 1:5 or 1:2, Sauve et al. 2000; Yin et al. 2002. The study by Lisin uses a solid:solution ratio of 1:20.
- It is not clear from the materials and methods if the solutions were shaken or agitated, but it is presumably that they were as the solution was changed and filtered after each processing cycle (every 4 days). Particle fragments can be seen (Fig 13) in the steel and lead shot treatment, but not so clearly in the others. Unfortunately, there were no controls[3] and no replicates in these experiments, so it is not clear if the fragments where the result of physical or chemical degradation. It has become common in experiments with nanomaterials to include inert physical control materials when attempting to establish the roles of either chemical or physical processes in ecological effects and this could have readily been undertaken in these experiments. It is obviously much less likely that physical agitation or collisions between the 'hard' steel shot and the soft lead shot would occur under field conditions one the material is deposited onto the soil surface.

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[2] This 'relevance' is specifically in relation to the behaviour and fate and steel and lead deposited onto shooting range soils in the form of shot.

[3] Physical influences of particles 'only' can be identified through the use of appropriate controls, e.g. Petersen et al. 2014.

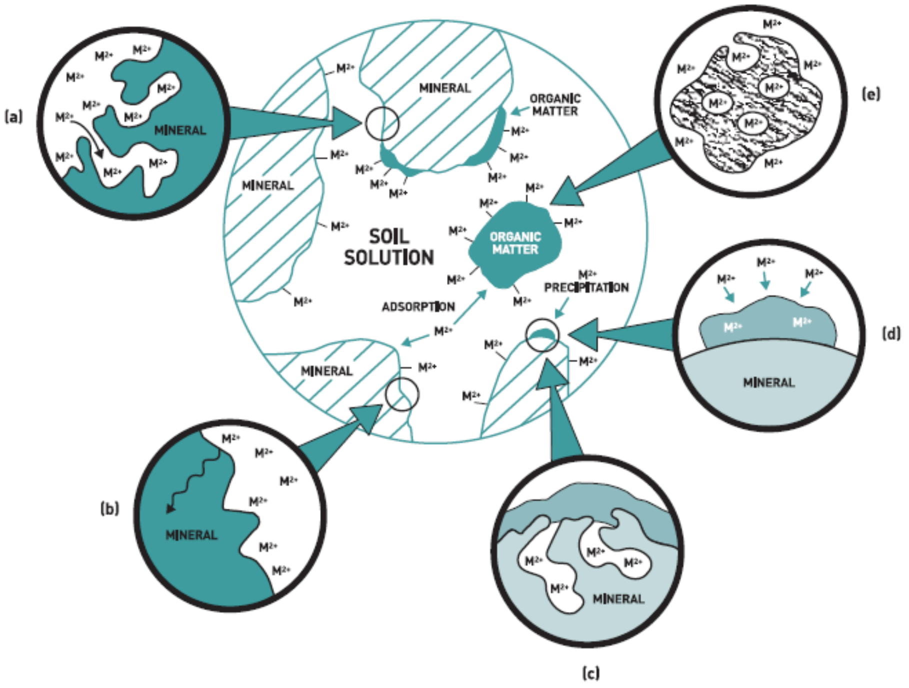

Figure 2.1 Adsorption, precipitation, and ageing processes in soil that may reduce the availability and transport of lead (ICMM 2007).

Iron is poorly soluble under typical soil conditions , being present in the form of hydrous oxide precipitates. These hydrous oxides of iron (and aluminium and manganese) in soils readily sorb trace element cations. Generally, these oxides of iron are poorly soluble under circumneutral and somewhat acid pH conditions, and these are identified by Lisin et al. (2022) as ‘suspended iron forms’. Importantly, under typical soil conditions these amorphous hydroxides of iron would be expected to age with time, becoming crystalline, less soluble, less sensitive to changing oxidation conditions of the surroundings and the co-precipitated metals (such as lead) would be less environmentally available (Smolders et al. 2009).

Under prolonged and extreme anaerobic conditions, some forms of amorphous iron oxides may solubilise, and the associated trace metals may be expected to be released. Experimentation in the field has demonstrated the importance of soil organic matter under these conditions, reducing solution trace element concentrations and environmental mobility (e.g. Dewey et al. 2021).

It is reasonable to expect steel shot to weather at a greater rate than lead shot in soils (ECHA 2020). However, evidence from the field and laboratory does not support the suggestions that mobile soluble iron compounds and complexes may result in ‘uncontrollable growth of the pollution of environment’ (presumably from both lead and iron). Under normal soil conditions, iron compounds will be of very low solubility, and the soil solution concentrations are also relatively low (<30 µg Fe L^{-1}). Studies at shooting ranges where both lead and iron are present, in large total quantities, demonstrate no evidence to support the suggestion of iron or lead movement through soils and into local ground and surface waters (e.g. Clausen and Korte 2009; Clausen et al. 2011; Barker et al. 2020).

Lisin et al. (2022) show a relatively large increase in the mean (and standard deviation) concentrations of suspended lead forms over the 25 cycles in the steel shot and lead shot mix for the atmospheric treatment (8.95 ± 8.35 µg L^{-1}) compared to the lead shot only treatment (1.23 ± 0.97 µg L^{-1}). As indicated by ECHA (2020), it is not clear why increased lead release would be expected in the presence of steel shot, and it certainly would not be expected to occur in a soil-system. The absence of suitable 'particle' or physical controls and the lack of experimental description in relation to agitation makes interpretation of this outcome extremely difficult. Barker et al. (2020) reviewed the speciation of lead and the weathering of shot from bullets (containing both lead and iron) at a shooting range in Alaska. The authors used similar scanning electron microscopy techniques to those of Lisin et al. (2022), but specifically examined the influence of iron from the soil and bullet in relation to lead behaviour. Barker et al. noted a close association between soil particles and the bullet weathering crusts, with lead mostly associated with the soil iron rather than the bullet sourced iron. In soils with pH > 5.2, lead mobility is largely precluded because of sorption and precipitation process. At soil pH values < 5.2, lead may theoretically become available, yet most soils have an abundance of iron, aluminium and manganese oxides that reduce lead mobility (Clausen et al 2011). As stated in ECHA 2020, the presence of iron in shooting range soils in which lead shot is present, which is lower than lead in the galvanic series, is not expected to enhance galvanic corrosion of the lead shot.

Reviewing the solution concentrations measured by Lisin et al. (2022) for the atmospheric treatment (rain), the mean dissolved concentrations of lead over the 25 cycles are five times lower in the steel and lead treatment (0.19 ± 0.08 µg L^{-1}) compared to the lead shot only treatment (1.02 ± 0.22 µg L^{-1}). Importantly, the lead released is noted to be in the form of suspended (particulate?) forms, yet it is the solution concentrations of metals, i.e. those that are readily exchangeable on soil surfaces and associated with dissolved organic carbon that are considered to be the most immediately environmentally relevant and potentially mobile in soils.

An increase in rates of weathering of lead shot, in the presence of steel shot is described in the solution experiments of Lisin et al. with a commensurate increase in steel shot weathering, and iron release, in the presence of lead shot. These outcomes are challenging to extrapolate to the field situations, where soil, climatic, and geogenic factors are likely to dominate chemical behaviour and fate. Indeed, reviewing the raw solutions data[4] provided in Lisin et al. for the total concentrations of iron released, over the 25 cycles (for the atmospheric precipitation), with and without lead shot present suggests a (just) statistically significant difference (P = 0.048) between these treatments, when <LoD (or LoQ, it is not clear in the paper) are substituted for ½ LoD. However, taking the dataset for the acid precipitation treatment; an apparently more extreme weathering scenario, there is no significant difference between these treatments regarding the total iron weathered and released to the solutions (P = 0.079). This suggests that the evidence of an increased pollution risk status from iron at shooting ranges associated with accelerated weathering of steel shot in the presence of lead shot, is

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[4] Unfortunately, only the meta data are presented in Table 2, and the raw mass data are not given in the supplemental material, or the details of the approach used to randomly select the shot or the balance apparatus used.

not clearly demonstrated by the relatively unrealistic exposure scenarios presented in these solution experiments.

There is little or no evidence in the scientific literature of increased transport of iron (or lead) in soils of shooting ranges (e.g. Almaroai et al. 2014; Hiller et al. 2021). Indeed, experimentation suggests that deliberately adding iron (albeit not necessarily in the form of steel shot) will reduce potential environmental risks from lead at shooting range sites from metal migration, through soils (Sanderson et al. 2012 cited in ECHA 2020), and certainly not reaching the concentrations in surface or groundwaters or occurring in the timeframes suggested by Lisin et al. (2022).

Speciation Modelling of Soil Porewaters

The scientific plausibility of increased lead release from shot over soils, in the presence of steel shot has been assessed through the use of speciation modelling. Modelled data were presented in ECHA (2020) that illustrated four scenarios associated with different pH and organic matter regimes. The scenarios modelled both represented essentially the same soil conditions but with either a very low (0 mg L^{-1} DOC) or very high (50 mg L^{-1} DOC) concentration of dissolved organic carbon (DOC) representing extreme conditions. It is not clear whether any organic matter was included in the solid phases, but as this was not noted it is assumed that the only organic matter present was in the dissolved (colloidal) phase. The scenarios also considered either an acidic (pH 4) or circumneutral (pH 7) pH in the soil porewater. These scenarios therefore represent the extremes of what is likely to occur in terms of the potential interactions between lead, iron, and organic matter.

The summary of the modelling scenarios is unclear about the proportion of iron that is present in a precipitated form as it notes the important soluble species, and for some of them notes that the species precipitates. The report also fails to distinguish between $FeOH^{2+}$ and $Fe(OH)_2^+$, although as both of these species precipitate to form iron hydroxide phases which can bind cationic Pb species the distribution of iron between these two species is unlikely to be particularly important. It is not clear whether the interactions between the dissolved inorganic lead species (e.g. Pb^{2+}) and the precipitated iron hydroxide phases that are formed has been taken into account, as the free ion activities of the relevant components were not reported. These issues have been further considered in additional speciation modelling simulations undertaken using a different speciation programme WHAM7 (version 7.0.5, CEHUK https://www.ceh.ac.uk/services/windermere-humic-aqueous-model-wham).

Iron is a redox sensitive metal, i.e. it can exist as either Fe(II) or Fe(III) depending upon the local conditions, and it is also subject to both complexation with dissolved organic ligands and precipitation as mineral phases. Whilst Fe(II) is generally considered to be the dominant form of iron under reducing conditions, and Fe(III) is generally considered to be the dominant form of iron under oxidising conditions, it is common for both forms to be present in natural soils and waters. It has been suggested that organic ligands may play a role in stabilising iron from redox processes (e.g. Hopkinson and Barbeau 2007), which suggests that the presence of thermodynamically unstable forms of iron (such as Fe(II) under oxic conditions) is most likely to be as complexes with organic matter. However, other studies have shown that Fe(II) is associated with precipitated $Fe(OH)_3$ minerals and can be oxidised under reducing conditions in some soils (Ahmad and Nye 1990). Consequently it is reasonable to assume that Fe(III) will be the dominant form of iron released from any dissolution of steel shot.

Here water chemistry conditions, that are broadly representative of soil porewaters were modelled using WHAM7 to investigate the effect of different conditions on lead availability, as indicated by the free lead ion activity in the solution. Any modelling of interactions with the solid phases of the soil are limited to particulate humic acid and particulate iron hydroxide minerals acting as binding phases for cationic species such as Pb^{2+}.

The conditions considered were pH values between 4 and 9 with both humic acid and $Fe(OH)_3$ in particulate forms, and both fulvic acid and $Fe(OH)_3$ in colloidal forms. The major ions in solution were represented by 1 mM concentrations of $CaCl_2$ and Na_2SO_4. Cu and Zn were also assumed to be present at a concentration of 0.1 M each to represent other trace elements that could compete with Pb for occupancy of binding sites on both colloidal and particulate organic (humic and fulvic acid) and inorganic (precipitated iron hydroxide) binding phases. The particulate $Fe(OH)_3$ was assumed to be associated with the soil material and the colloidal $Fe(OH)_3$ was assumed to be associated with the added iron from steel shot. Iron added to the soil porewater from the corrosion of steel shot was assumed to be in the form of Fe(III) because Fe(II) is only likely to be stable under reducing conditions. However, once in a precipitated form the source of the iron is not important. The colloidal $Fe(OH)_3$ was allowed to precipitate from a quantity of Fe(III) present in the solution, and was not specified as having a concentration at the start of the speciation calculations other than the quantity of Fe(III) in solution. Variation was considered in the concentrations of particulate humic acid, particulate iron oxides, colloidal fulvic acid, total Fe(III), total Pb, ionic strength, and partial pressure of CO_2.

The precipitation of any added iron (e.g. from steel shot) as colloidal $Fe(OH)_3$ is very limited in acidic soils, i.e. with a pH below 7, and is also reduced by high concentrations of other binding phases such as particulate humic acid, colloidal fulvic acid, and particulate $Fe(OH)_3$. In the calculation of Pb speciation the WHAM model does not distinguish between lead that is bound to either particulate or colloidal iron oxide phases. Figure 2.2 shows the quantity of Pb bound to the various important binding phases (i.e. humic acid, fulvic acid, and iron oxides) as a function of the free ion activity of the $Pb^{2+}_{(aq)}$ ion, i.e. $\{Pb^{2+}\}$. The quantity of Pb bound to these binding phases is expressed as the ν-value (nu value) which is the number of moles of Pb bound per g of binding phase. This shows that on a mass basis iron oxide precipitates are a much more important binding phase for dissolved Pb species than the organic humic and fulvic acid phases under virtually all of the conditions considered by the modelling scenarios.

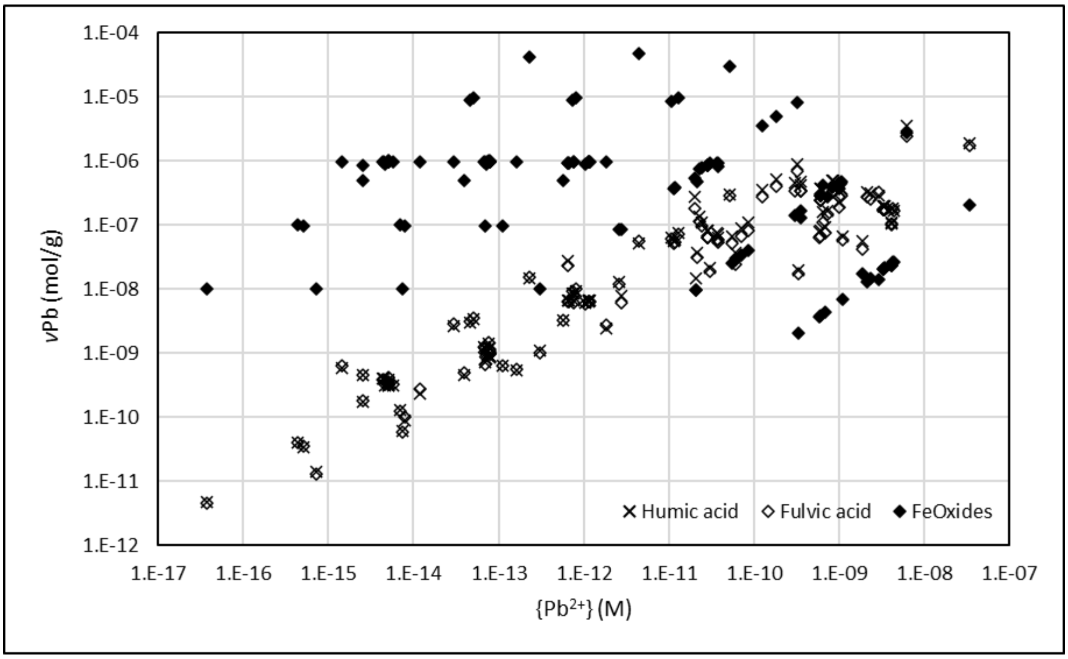

Figure 2.2 Quantity of Pb associated with different binding phases as a function of Pb^{2+} activities in solution

Further calculations were also performed to simulate titration of the soil solutions with additional iron from added steel shot in order to evaluate the effect that this is likely to have on the availability (mobility and bioavailability) of Pb in the soils. At very low levels of iron, which may be considered as representative of scenarios without addition of steel shot, the free ion activity of Pb^{2+} in the soil porewater is effectively governed by pH (Figure 2.3). As iron is added to the solution, from the corrosion of added steel shot, there is a decline in the free ion activity of Pb^{2+}, i.e. the potential availability and toxicity of lead is reduced by additions of iron, except at low pH, where a slight increase in the Pb^{2+} activity is observed which is likely to be due to increased competition for binding to fulvic acid from Fe^{3+}.

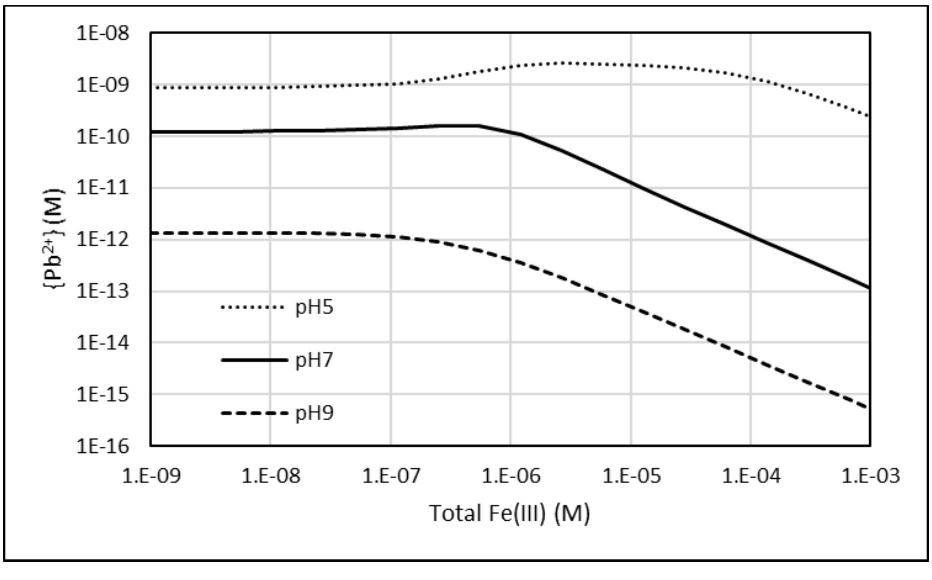

Figure 2.3 Changes in the free ion activity of Pb with increasing Fe(III) concentrations in porewater

The increase in Fe(III) concentrations in soil porewater from corrosion of steel shot results in the precipitation of colloidal $Fe(OH)_3$ (Figure 2.4), which also acts as an additional binding phase for Pb^{2+}. In these simulations no other sources of iron oxide or hydroxide phases were assumed to be present, which explains the apparent increase in free Pb^{2+} activities under low pH conditions because the only available binding phase under those conditions in these simulations is assumed to be fulvic acid. Under low pH conditions (pH 5) iron hydroxide colloids are not precipitated at appreciable levels until total iron concentrations exceed 10 mM, but additional colloidal Fe(OH)3 binding phases are expected to be formed at much lower concentrations of added iron under higher pH conditions.

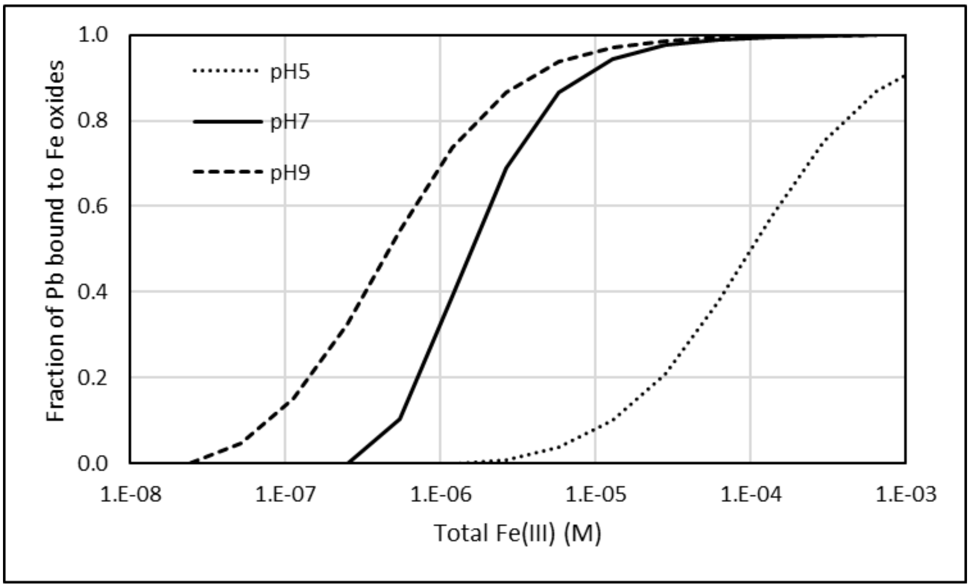

Figure 2.4 Changes in the fraction of Pb bound to precipitated iron oxides with increasing Fe(III) concentrations in porewater

Figure 2.5 shows the changes in the fraction of Pb associated with fulvic acid under these conditions with increasing iron concentrations. This figure explains the fate of Pb under those conditions where Pb is not complexed by precipitated colloidal iron hydroxide. Under these conditions the majority of the Pb is complexed by fulvic acid, rather than being present in a potentially bioavailable inorganic form. As the concentration of precipitated colloidal iron hydroxide increases due to the addition of iron from the corrosion of steel shot the complexation of Pb by the iron hydroxide becomes a more important binding phase than the fulvic acid. However, under acidic conditions where iron hydroxide does not tend to precipitate there is some displacement of Pb from the fulvic acid due to competition for binding from Fe^{3+}.

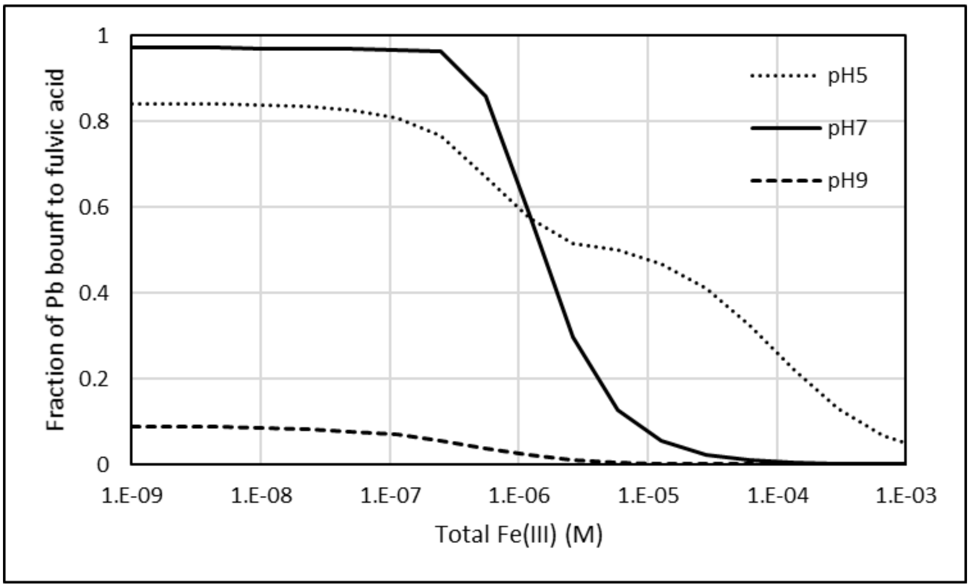

Figure 2.5 Changes in the fraction of Pb bound to fulvic acid with increasing Fe(III) concentrations in porewater

All of these speciation calculations ignore the possibility of additional binding phases in soil due to other metal oxide or hydroxide phases, such as aluminium or manganese, and additional mineral binding phases such as silica, quartz, or clay minerals. These simulations are therefore simplified considerably compared to the conditions present in natural soils, but importantly assume that these further binding phases are absent and are therefore not available to complex Pb (i.e. they are likely to be conservative predictions in relation to sorption of Pb from solution). The titration simulations also assume that the only organic components are colloidal fulvic acid in the solution and that the only other binding phase is due to the precipitation of iron hydroxide from dissolved Fe(III), whereas in reality there will already be iron hydroxide mineral phases present in the soil. These calculations are therefore conservative with respect to the availability of Pb, but demonstrate the likely implications of the addition of iron on Pb availability in soils more clearly.

Under circumneutral or higher pH conditions in soil porewater where total concentrations of Fe(III) exceed approximately 10 µg L^{-1} a significant proportion of the iron present would be expected to form colloidal $Fe(OH)_3$ precipitates, which acts as a binding phase for any Pb species that are present in the porewater solution. Under low pH conditions Fe(III) precipitation is much more limited and dissolved Fe(III) could potentially displace a small amount of Pb from binding sites on fulvic acid, although the displaced Pb would be likely to become associated with other binding phases present in the soil.

Overall, these model simulations suggest that the addition of iron from the corrosion of steel shot would be likely to reduce, rather than increase, the availability and mobility of Pb already present in soils.

Iron added to soils from the corrosion of steel shot is likely to be present as Fe(III), which tends to precipitate as $Fe(OH)_3$ and can then act as an additional binding phase for Pb in the soil. Under acidic conditions iron is less likely to form precipitates that can reduce the availability of Pb, although under these conditions both Pb and Fe are more likely to be mobilised from shot particles into the soil matrix.

3 CONCLUSIONS

- The study by Lisin et al. (2022) presents results from solution-based experiments with steel and lead shot, extrapolating the findings to soil-scenarios and hypothesising several relatively extreme environmental outcomes in relation to lead and iron behaviour.
- The experimentation lacks environmental relevance and could reasonably be suggested to be of ambiguous reliability in relation to the lack of appropriate controls, replicates, and reporting.
- It is possible that steel shot will weather at a greater rate in the soil environment, than lead shot (as indicated in ECHA 2020).
- Evidence presented by Lisin et al al. of lead shot weathering in solutions at a greater rate in the presence of steel shot is somewhat confounded by the lack of suitable controls in the testing solutions
- The suggestion that steel shot weathering is greater in the presence of lead shot is equivocal. The solution iron concentration data suggest this may not be a statistically significant effect for all the treatments. Further, the absence of the raw mass measurements means this cannot be verified, as only meta data are presented.
- Field-based evidence does not support the claims suggested in the paper by Lisin et al. regarding accelerate lead migration or iron impacts upon surface and ground waters.
- Chemical speciation modelling indicates that additions of iron into the soil porewater will reduce lead availability due to the formation of precipitates that lead binds to, except under acidic conditions where iron does not precipitate readily. The pH buffering capacity in the synthetic solutions used by Lisin et al. is negligible compared to what would be expected to be present in soils, meaning pH changes observed in solutions are unlikely to be replicated in the field.
- Where iron hydroxide precipitates are present, they are a more important binding phase for lead species than organic matter.
- Organic matter is an important binding phase for lead species where iron hydroxide precipitates are absent.
- The binding of lead species to organic matter or iron hydroxide precipitates reduces the potential for lead to be mobilised or cause toxicity.
- The chemical speciation modelling demonstrates that the claims made by Lisin et al. are unlikely. However, it is possible that under acidic conditions, where iron is not significantly precipitated, that a very small increase in lead availability could occur due to displacement from organic binding phases by iron, although the displaced lead would be likely to become associated with other binding phases present in the soil.

The outcome of this review of recent data suggests no changes are required in the conclusions given from ECHA's previous assessments.

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