



Predicting and assessing the potential ecological impact on the Wyre Estuary of the surfactant, EEA-NH₄, contained in the final effluent from AGC Chemicals Europe, Ltd.

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1 EXECUTIVE SUMMARY

Over the last year AGC Chemicals Europe Ltd (AGCCE) have commissioned three studies in support of their discharge consent into the Wyre estuary. This report is focussed particularly on the potential dispersal and impacts of EEA-NH₄ which is used as a surfactant on site.

The four studies commissioned were:

- Hydrodynamic modelling to predict the dilution and dispersion of EEA-NH₄
- Hydrodynamic modelling to predict the dilution and dispersion of EEA-NH₄ based on a discharge concentration figure used by the Environment Agency in their modelling assessment.
- A direct toxicity assessment of the final effluent from the AGCCE outfall.
- An ecological survey of the macrobenthos and saltmarsh of the Wyre estuary in the vicinity of the site.

Hydrodynamic Modelling

The hydrodynamic modelling predicted the dilution and dispersion of EEA-NH₄ within the estuary. The results were then compared to the PNEC (a value provided by AGCCE) for marine organisms to determine whether any toxicity would be predicted in the marine environment.

The modelling showed that concentrations of EEA-NH₄ had reached equilibrium after 8 tides from first being released. Using the mean discharge load from a 5-year period, the PNEC was exceeded in an area of just under 10 hectares.

Within the 5 year period 97% of the results were within 3 standard deviation results of 0 mg l⁻¹ to 6.39 mg l⁻¹ from the average of 2.24 mg l⁻¹. The maximum EEA NH₄ concentration result from the 5 year period was 8.5 mg l⁻¹. The PNEC has been derived using the standard guidance from ECHA taking into account the uncertainties in the data.

Direct Toxicity Assessment

The direct toxicity assessment (DTA) determined the acute toxicity of the final effluent from the AGCCE outfall to marine alga and copepods.

DTA takes into account any synergistic, additive or antagonistic effects of the individual contaminants within the effluent. The result was then used together with the dilution from the modelling study to predict whether any toxicity would be predicted in the receiving waters.

The worst-case prediction from the DTA study demonstrated that at the end of the mixing zone as defined by the model (100 metres upstream and downstream from the AGCCE outfall), there would be an estimated acute effect in the receiving water at the edge of the mixing zone during states of the tide with this dilution.

However, this minimum dilution will only occur over low tide and, as a result, any organisms present would have a relatively short exposure period which is likely to reduce this toxicity. No toxicity was predicted if the generic model from the H1 guidance was used.

Ecological Survey

The ecological survey showed that concentrations of EEA-NH₄ have declined in the sediments since 2016 and been below detectable concentrations since 2018. The ecological survey measured actual concentrations of EEA-NH₄ in the sediments and also determined whether the effluent had any impact on the macrobenthos or saltmarsh in the vicinity of the outfall.

There was no evidence from the ecological survey conducted of any impact of the effluent from the site on either the benthic macrofauna of the estuary or on the saltmarsh in 2021. Rather, the chief factor that determines the benthic community structure within the estuary is sediment granulometry. This conclusion is based on the analysis of the results from the biennial surveys of the estuary that have been undertaken since 1996. In these analyses, the stations were grouped according to their sediment granulometry using Principal Component Analysis before the benthic macrofauna were compared between stations using multi-variate statistical techniques. It has therefore been possible to identify the dominant factors that determine the community structure within the estuary, and, by effectively controlling for sediment granulometry, determine whether there are any local anthropogenic impacts.

2 INTRODUCTION

2.1 The Hillhouse International Site complex

Since the fragmentation and decline of the ICI chemical complex in the 1980s and 1990s, chemical production at the Hillhouse International site at Thornton Cleveleys has reduced considerably. It is now a 138-hectare site, and following the closure of Vinnolit in 2020, only two principal companies continue to manufacture chemicals on site and discharge into the estuary via the main outfall. These are AGC Chemicals Europe Ltd. and Victrex Plc that produce fluoropolymers (PTFE and ETFE), and high-performance plastics respectively. The aqueous effluents produced by these two companies are discharged through an outfall, via separate chambers.

2.2 Dilution and dispersion studies

The 3-dimensional hydrodynamic and water quality model ^[1] was updated and used in 2021 to assess the potential releases, distribution and concentrations of the surfactant EEA-NH₄ through the AGCCE effluent stream to the estuary ^[2]. It was necessary to update the model as it is known that the main channel in the vicinity of the outfall can fluctuate from one side of the estuary to the other ^[3]. By updating the bathymetric data, the most recent position of the channel could be identified and also the effect of temporal changes in the channel could be inferred by comparing the predictions with the original model with those from the updated version.

2.3 Toxicity of the effluent from the AGCCE outfall.

The toxicity of the effluent from the AGCCE outfall has been assessed using Direct Toxicity Assessment (DTA) ^[4] on several occasions. The initial study was part of an Integrated Pollution Prevention and Control (IPPC) application for the site undertaken in 2005 ^[5]. Following concerns over the persistence of the surfactant used at the time Ammonium Perfluorooctanoate (APFO), AGCCE undertook an environmental scoping exercise investigating the potential benefits of a change ^[6].

In 2009, AGCCE assessed the potential effect on the site's final effluent of changing the surfactant they used in their manufacturing process from APFO to difluoro[1,1,2,2-tetrafluoro-2-(pentafluoroethoxy) ethoxy] acetate (EEA-NH₄) by determining the ecotoxicity of site effluent spiked with the new surfactant ^[7].

After this change, in line with the Environment Agency guidance for the use of DTA in PPC Impact Assessments, further DTA of the site final effluent was undertaken in 2011 ^[8]. Following improvements made at the plant after the adoption of EEA-NH₄ as a surfactant, and studies being completed for the Environment Agency, the toxicity of the effluent from the AGCCE outfall (which included octylphenolethoxylate (OPE)) was re-assessed in 2021 ^[9].

2.4 Ecological impact assessments of the discharges from the Hillhouse site

Modelling individual contaminants produces data that can be compared with established Environmental Quality Standards (EQS) and both acute and chronic toxicity data. A prediction can then be made on whether the modelled concentrations are likely to cause acute or chronic impacts in the environment together with the area over which these effects may be experienced. However, these predictions are often based on a worst-case scenario (e.g., using maximum consented flow rates and concentrations, worst case dilutions, and toxicity data that has a safety factor in it) and do not generally consider the toxicity data for the whole effluent. This can to a certain extent be mitigated by using data from tidal averages and from DTA.

Similarly, the results from DTA are used to predict the toxicity of the whole effluent in the receiving waters. For the AGCCE effluent this is based on the toxicity of the effluent to marine algae and copepods. The derived toxicity data is then used to determine a Maximum Acceptable Threshold Concentration which is then compared to the Predicted Environmental Concentration (PEC) to determine whether any effects are predicted. The PEC is derived using generically available dilution data for estuaries from H1 ^[10] or from site specific dilution data from hydrodynamic modelling. As with the modelling, it is important to note that these are generally worst-case predictions using the most toxic sample taken.

The final arbiter is the environment itself. Ecological impact or improvement in estuaries and coastal waters can best be determined by monitoring changes in the diversity and abundance of the invertebrate fauna that inhabit their sediments ^[11]. These studies have been carried out from 1979 to the present with the most recent survey being undertaken in 2020 ^[9]. Physico-chemical data is also collected during these surveys and the data assessed

against any established toxicity guidelines. It is also compared to the benthic community data to determine if the concentrations observed are affecting the macrofaunal community structure over the survey area.

Collectively, these now biennial benthic ecology surveys form a continuing ecological monitoring programme in the Wyre estuary. The results provide a robust baseline, against which any present or potential impacts from future developments, or improvements associated with the discharge of effluent from the Hillhouse site, can be assessed. It also defines the areas of impact where these are present.

2.5 Ecological importance of the Wyre estuary

The Wyre estuary is one of the components of the Morecambe Bay Special Protection Area (SPA) and flows into the Morecambe Bay Special Area of Conservation (SAC). It also forms part of the Wyre Lune Marine Conservation Zone (MCZ) which was created primarily to protect breeding populations of smelt *Osmerus eperlanus* [12].

The area covered by the SPA is designated as a Wetland of International Importance under the Ramsar Convention (Ramsar, Iran, 1971). The site (and estuary) is also notified as a Site of Special Scientific Interest (SSSI) under the UK Wildlife and Countryside Act, 1981 and the UK Countryside Rights of Way Act, 2000. The primary focus of these designations is the protection of the internationally important populations of waterfowl that occur within the SPA and SSSI.

As well as protected bird life, the estuary also supports a number of other habitats and species which are considered to have high conservation value. Many of the fish species that occur in the estuary are UK species of principle importance and the estuary also supports European eel which are on the IUCN red list as critically endangered. Habitats of principle importance that occur within the Wyre estuary include coastal saltmarsh and inter-tidal mudflats. The estuary also supports recreational fisheries for marine fish as well as trout, salmon and eel and is an important local amenity. The estuary and coastal waters also support commercial fish and shellfish fisheries.

3 SCOPE

This report aims to collate the key results and conclusions from the four studies carried out over the last year, in relation to predicting and assessing the ecological impacts of EEA-NH₄ which is released in the final effluent from AGCCE.

These were:

- Hydrodynamic modelling to assess the potential impacts from releases of EEA-NH₄ [2]
- Hydrodynamic modelling to predict the dilution and dispersion of EEA-NH₄ based on a discharge concentration figure used by the Environment Agency in their modelling assessment.
- Direct Toxicity Assessment of the final effluent from AGCCE outfall to determine the toxicity of the effluent and therefore whether toxic effects were predicted [9]

- Ecological impact assessment of the benthic macrofauna, the saltmarsh and the physico-chemistry of the sediments to determine whether there is any evidence of an impact caused by the effluent from the site and, if so, determine the extent of any such impact [3].

4 METHODS

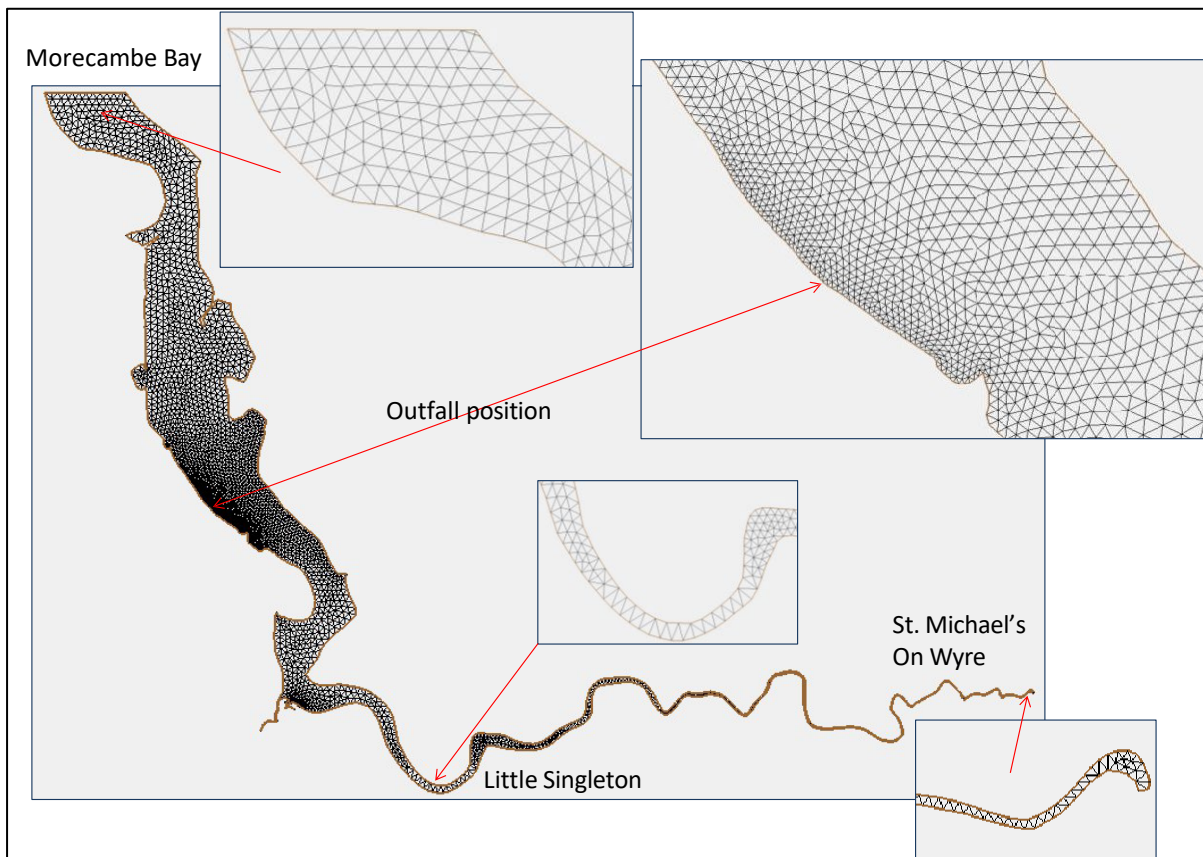
Full details of the methods employed in each study are provided in the original reports [2, 3, 9, 18]. Very brief summaries are provided in the sections below.

4.1 Hydrodynamic modelling

The Wyre model covers an area from Morecambe Bay southwards up the estuary of the Wyre and eastwards to St. Michael's on Wyre which is the limit of the tidal influence (Fig. 1). It was built on the FVCOM [13] software and consists of a 3-dimensional (3-D) tidal hydrodynamic model coupled with a 3-D water quality model. The hydrodynamic model computes tidal currents, salinity and temperature on a 3-D grid over the area simulating the variation of these parameters in the horizontal plane and their vertical variations. The water quality model is fully linked to the hydrodynamic model and is set up to compute chemical concentrations across the area resulting from effluent discharges at specified locations. The software was enhanced at Ecospan to compute tidal average and tidal maximum concentrations as well as concentrations at different stages of the tidal cycle.

The model used effluent flow and EEA-NH₄ concentration data from 2016 to 2020 as the basis of the predictions. Using the model, predicted EEA-NH₄ concentrations, for both the average discharge and maximum discharge loads were calculated for both spring and neap tides.

Fig. 1. Area covered by the model and the grid structure used.



4.2 Direct toxicity assessment

In order to assess the variability of the effluent from the AGCCE outfall, 6 samples were tested. This was done by using one spot sample and one 24 h composite sample taken over a three-week period in February 2021. The combination of spot and composite samples were used to ensure that both the day-to-day effluent toxicity was captured, along with any potential short-term variability.

As the effluent discharges to the marine environment, toxicity testing was undertaken to the marine diatom *Skeletonema costatum* and the copepod *Tisbe battagliai*. The tests used during this investigation were in compliance with those recommended in the DTA technical guidance documents [14, 15] and carried out by Ecospan Environmental Ltd which is MCerts approved for DTA testing.

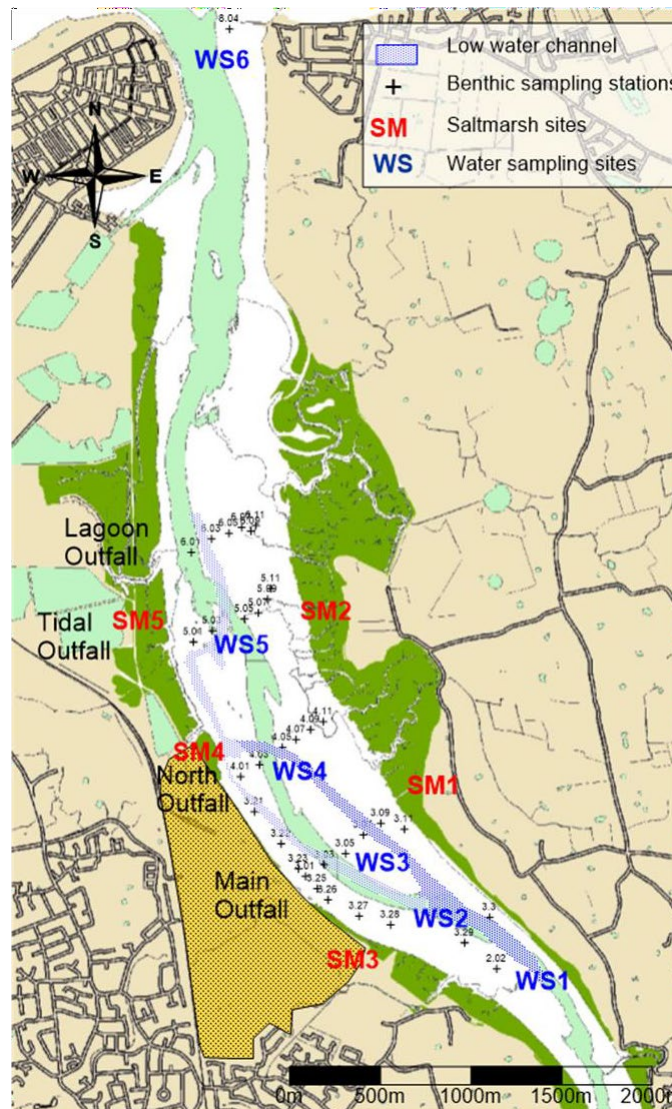
4.3 Ecological impact assessment of the benthic macrofauna and saltmarsh of the Wyre estuary

A survey of the benthic macro-invertebrate fauna inhabiting the sediments of the Wyre Estuary and the health of the saltmarsh was carried out on spring tides in June 2020.

The survey followed the revised standard survey design used since 1996. Thirty-five stations were located along 4 transverse transects running across the estuary, from the west bank to the east bank and an additional longitudinal transect near the Main Outfall running parallel to the low water channel. Two reference stations, stations 8.04 and 2.02 marked the

downstream and upstream limits of the survey area respectively. Water samples were taken at approximately mid channel positions at high water from six locations in the survey area for fluoride analysis. Five areas of saltmarsh were also assessed. The sampling locations are shown on a chart in Fig. 2.

Fig.2. Ecological impact assessment station positions and channels



Stations were sampled using a 0.0625 m² quadrat, which was excavated to a depth of 10 cm and the sediment washed through a 1 mm mesh sieve. The fauna and other material retained on the sieve were then preserved in a solution of 10% buffered formalin in seawater.

At each station, a sample of undisturbed sediment was collected for sediment particle size analysis (PSA) and sediment fluoride analysis. Further samples were taken for EEA-NH₄ analysis from stations 3.01, 3.03, 3.22, 3.23, 3.25, and station 8.04.

Five areas of saltmarsh identified as the most likely areas to be affected by any potential waterborne contamination from the site due to their proximity were surveyed. The survey followed the appropriate methods recommended in guidelines developed by the Joint Nature Conservation Committee [16].

Two statistical approaches were applied during the analysis of the data. Simple, uni-variate statistics such as mean number of taxa per sample, mean number of individuals per sample, mean proportion of sediment in each particle size band, and diversity/equitability indices were calculated and compared for each station. Additionally, community analysis techniques ^[17] were used to compare the stations and provide a measurement of overall similarity between the stations, both in terms of their constituent taxa and relative abundance and the physico-chemical composition of their sediments.

5 RESULTS AND CONCLUSIONS

5.1 Hydrodynamic modelling

Hydrodynamic modelling was undertaken to predict the dilution and dispersion of EEA-NH₄ based on a discharge concentration figure (3.2 mg l⁻¹) used by the Environment Agency in their modelling assessment.

This was completed against the minimum, average and maximum flow rates of 17.33, 34.8 and 44.22 m³ hr⁻¹. The EEA NH₄ loads discharged to the estuary for each scenario were calculated from the data, and the concentrations in the estuary were predicted for both the spring and neap tides using the Ecospan model. Annual average estimates of concentrations and areas exceeding the PNEC were calculated as the average of the spring and neap results.

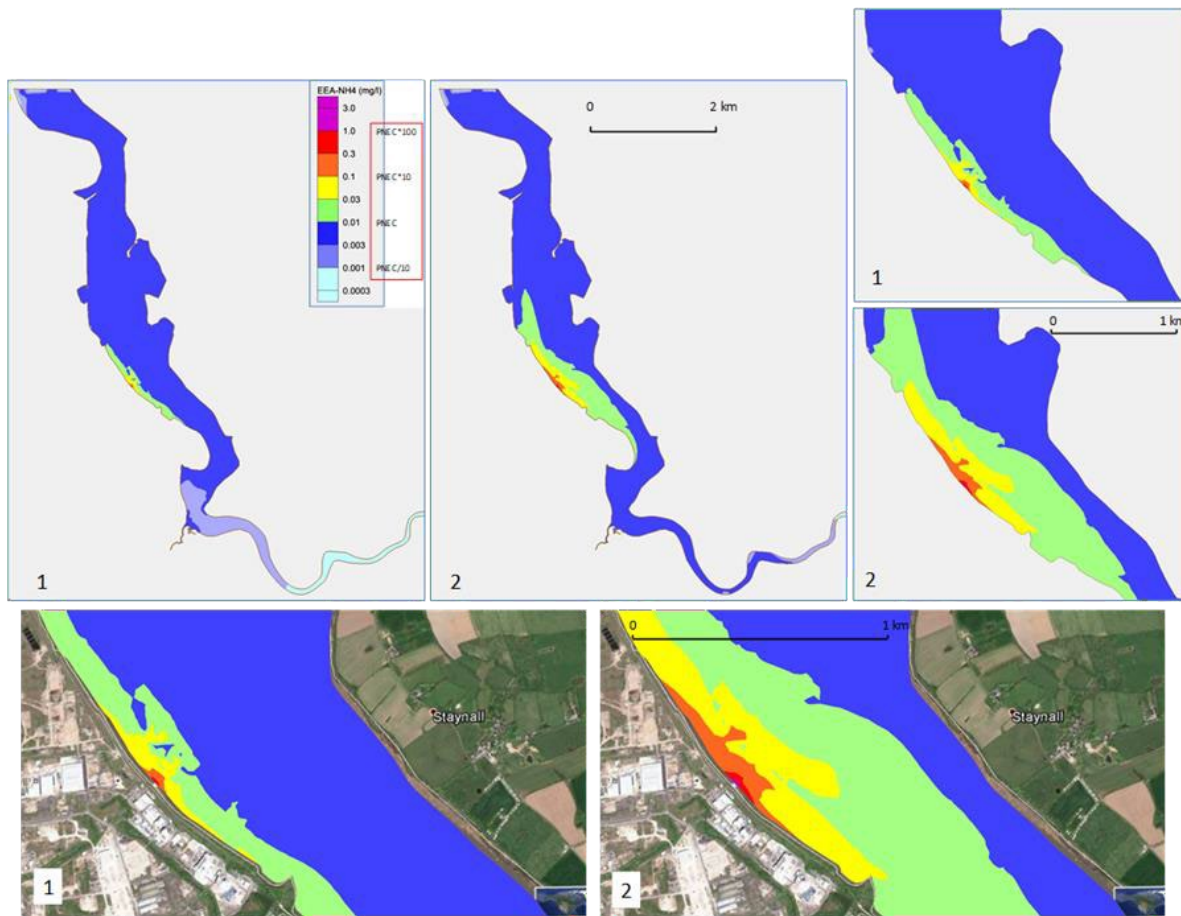
The PNEC was derived from toxicity data on freshwater species representing three trophic levels using the standard guidance published by ECHA in the context of the REACH Regulation ^[19]. The lowest PNEC from the acute tests was divided by a safety factor of 1000 to account for uncertainties between acute and chronic effects and by a further factor of 10 to account for uncertainties about differences in sensitivity between freshwater and marine species. Note: an option to consider in the future is that ecotoxicity data specific to marine organisms is collected, so to enable this assessment to be refined.

The results from the hydrodynamic modelling showed:

- For the minimum flow (and load) the peak concentrations at the upstream and downstream ends of the mixing zone are above the PNEC, while the peak concentrations at Shard Bridge, Fleetwood and the Wyre Light are below the PNEC. The area exceeding the PNEC was 6.1 ha on tidal average.
- For the average flow (and load) the peak concentrations at the upstream and downstream ends of the mixing zone and at Fleetwood and the Wyre Light are above the PNEC, while the peak concentration at Shard Bridge is below the PNEC. The area exceeding the PNEC was 14.2 ha on tidal average.
- For the maximum flow (and load) the peak concentrations at the 5 locations are above the PNEC. The area exceeding the PNEC was 20.1 ha on tidal average.

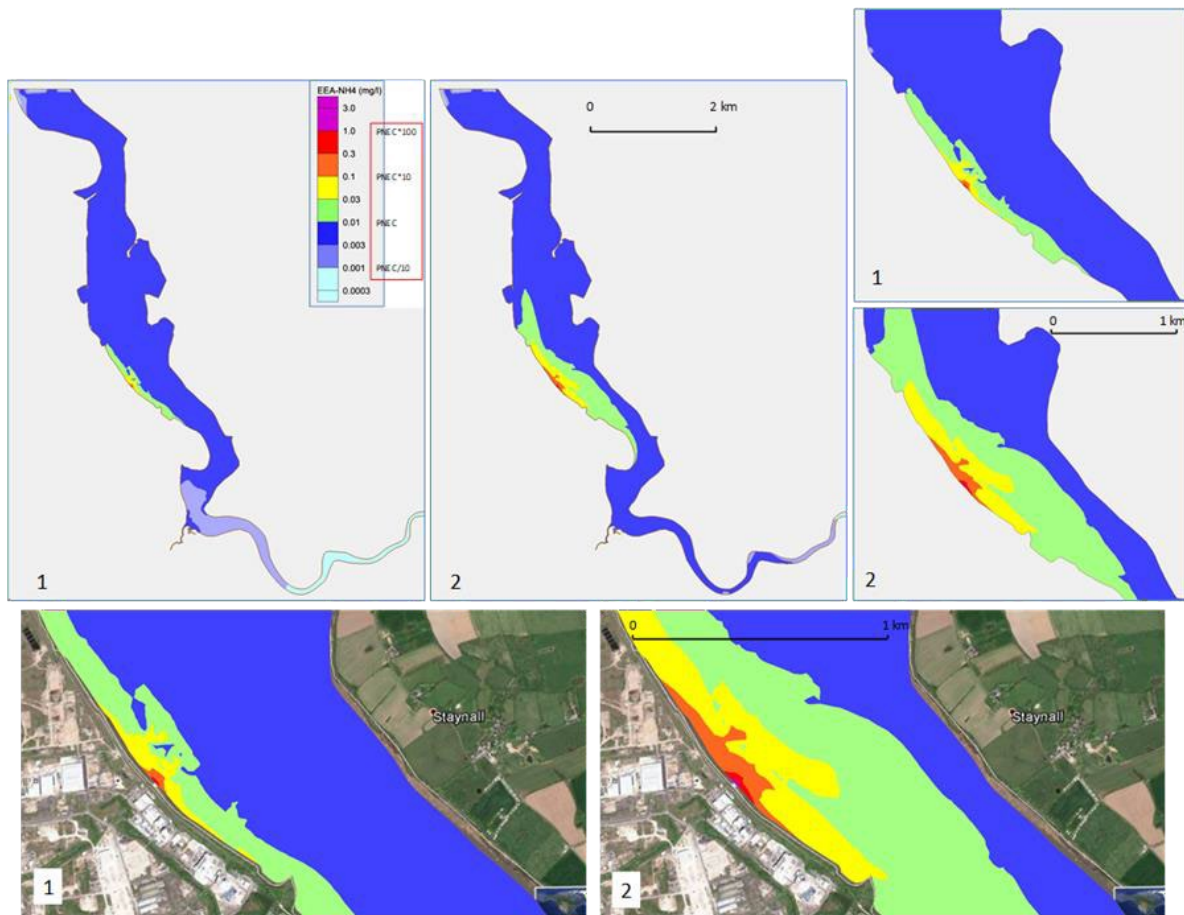
The predicted tidal average and tidal maximum concentrations (mg l⁻¹) of EEA NH₄ in the Wyre estuary for the average discharge load scenario (spring tide) are shown in Fig. 3.

Fig. 3. Predicted tidal average (plots labelled "1") and tidal maximum (plots labelled "2") concentrations (mg l⁻¹) of EEA-NH₄ in the Wyre estuary for the average discharge load scenario: spring tide.



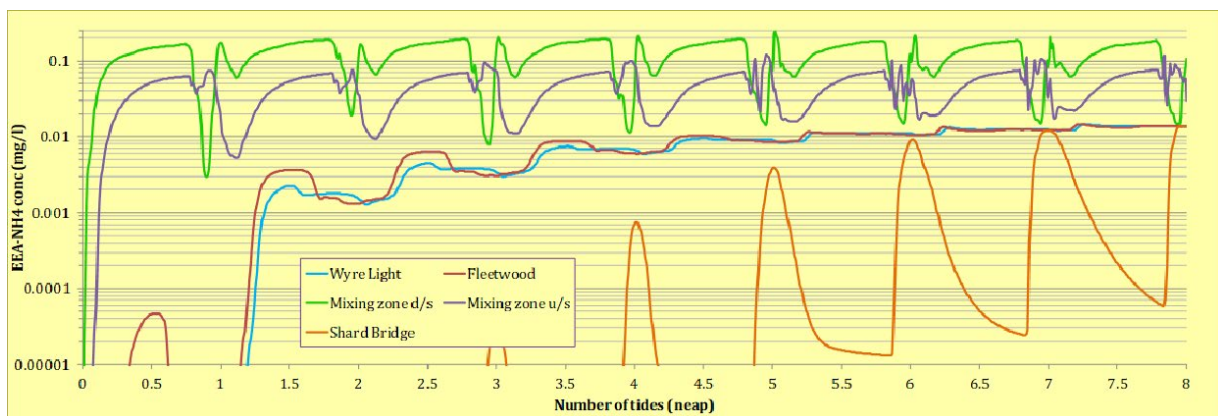
The predicted tidal average and tidal maximum concentrations (mg l⁻¹) of EEA NH₄ in the Wyre estuary for the average discharge load scenario (neap tide) are shown in Fig. 4.

Fig. 4. Predicted tidal average (plots labelled "1") and tidal maximum (plots labelled "2") concentrations (mg l⁻¹) of EEA-NH₄ in the Wyre estuary for the average discharge load scenario: neap tide.



Since EEA-NH₄ is relatively stable, the model runs were extended to see at what point the loading in the estuary reached an equilibrium. It can be seen from Fig. 5. That this is largely achieved after 8 tides over neap conditions (worst case).

Fig. 5. Predicted time series of EEA-NH₄ concentrations (mg l⁻¹) at different locations using the average load over neap tides.



5.2 Direct toxicity assessment

The overall ecotoxicity of the AGCCE effluent to marine species was observed to be low with Maximum Allowable Threshold Concentrations (MATCs) in the range of 3.6 to 7.1% for the marine copepod *Tisbe battagliai* and 7.1% to over 10% (the lowest dilution tested) for the marine alga *Skeletonema costatum* (Table 1). (The MATC is defined as the geometric mean between the No Observed Effect Concentration (NOEC) and the Lowest Observed Effect Concentration (LOEC)).

Table 1. Summary toxicity statistics for AGCCE's effluent as tested in 2021.

Algal Biomass 72h results (%)				Tisbe 48h results (%)			
NOEC	LOEC	EC50	MATC	NOEC	LOEC	EC50	MATC
10.0	>10.0	>10.0	>10.0	2.5	5.0	7.6	3.6
10.0	>10.0	>10.0	>10.0	2.5	5.0	>10.0	3.6
10.0	>10.0	>10.0	>10.0	5.0	10.0	>10.0	7.1
10.0	>10.0	>10.0	>10.0	5.0	10.0	>10.0	7.1
10.0	>10.0	>10.0	>10.0	2.5	5.0	5.6	3.6
5.0	10.0	>10.0	7.1	2.5	5.0	6.2	3.6

The toxicity of the effluent in 2021 was within a similar range to that observed in the six samples tested in 2011 (MATC between 1.8% to >10 % for *Tisbe battagliai* and greater than 10% for all algal tests) [8]. However, these results show that the effluent is less toxic than that tested in 2005 prior to the use of EEA-NH₄. (MATC of between 2.4 – 5.7 % for algal biomass and 1.79 – 5.7 % for *Tisbe battagliai*) [5].

If the generic approach following the H1 Integrated Pollution Prevention and Control (IPPC) guidance [10] is used, the effluent dilution can be calculated by the equation:

$DC = \frac{EFR}{DR}$ where DC= Diluted concentration, EFR = effluent flow rate (m³/s) and DR= Dilution rate (for high flow estuaries a value of 15 is provided by the guidance).

For this effluent the following calculation is $DC = 0.009667/15 = 0.00064$ which provides a PEC of 0.0644 %. Using the lowest MATC from the tests conducted in 2021 gives a PNEC of 3.6%. Comparing the two values, PEC/PNEC we get a ratio of 0.018. This means that no acute toxicity would be predicted in the receiving environment. An effect would only be predicted if the ratio is > 1 and therefore there is currently a 55 times safety factor using this assessment approach.

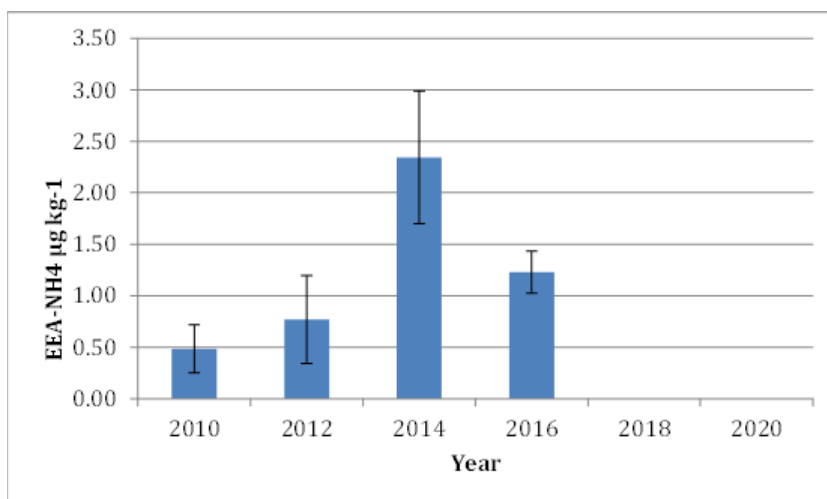
The estuary has been modelled using the site-specific dilution data. In that case, the worst-case dilution of the effluent at the edge of the mixing zone (10-fold) provided by the hydrodynamic modelling gives a PEC of 10%. Using the MATC generated by the most toxic sample in these tests in 2021 (3.6 %), this gives a PEC/PNEC ratio of 2.8. This suggests that an acute effect would be predicted in the receiving water at the edge of the mixing zone during states of the tide with this worst-case dilution. However, this minimum dilution will only occur over low tide and, as a result, any organisms present would have relatively short exposure period which may reduce any ecotoxicity that would potentially be predicted.

5.3 Ecological impact assessment of the benthic macrofauna and saltmarsh of the Wyre estuary

This long-term monitoring project has consistently shown that the concentrations of contaminants (including EEA-NH₄, where present in measurable levels) are associated with the sediment granulometry. Since the sediment granulometry is in turn heavily influenced with each station's position relative to the channel, fluctuations in the position of the channel can substantially alter the sediment granulometry (and hence contaminant concentrations) within the survey area. Additionally, improvements made at the plant will also be a contributing factor in reducing the concentrations of EEA-NH₄ in the sediments.

Concentrations of EEA-NH₄ measured during the biennial surveys since the start of its use in 2010 have shown that from 2010 to 2014 concentrations in those sediments around the outfall and the reference station at 8.04 increased from a mean of just under 0.50 µg kg⁻¹ to just approximately 2.3 µg kg⁻¹. However, in 2016, mean levels dropped to approximately 1.5 µg kg⁻¹ and have not been at detectable levels since (Fig. 6). The data shows that EEA-NH₄ is not accumulating in the sediments of the Wyre estuary, which is to be expected due to the high water solubility and low log K_{oc}. It is noted that the lack of detectable levels from 2018 onwards corresponds with an unavoidable change in the analytical laboratory, however the limit of detection (1 µg kg⁻¹) currently achieved is more sensitive than that achieved previously and would be sufficient to show EEA-NH₄ contamination if it was present in similar concentrations to previous years.

Fig. 6. Mean concentrations of EEA-NH₄ from 2010 – 2020 (+/- S.E.).

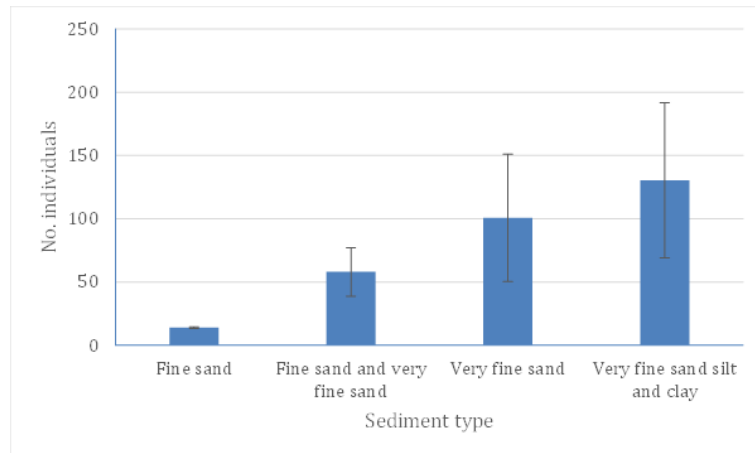


It should be noted that even in 2014, when the highest levels were observed, the peak concentration at any station was only half that of the PNEC for marine sediment dwelling organisms ^[2] of 10 µg kg⁻¹. (This PNEC was derived from freshwater fish and invertebrate data by applying a safety factor). It is therefore very unlikely that this compound is having any impact on the benthic macrofauna of the Wyre estuary, even at those stations very close to the outfall.

Results from the uni-variate and multi-variate statistical analysis of the benthic macrofauna data show that the dominant factor in determining community structure within the survey

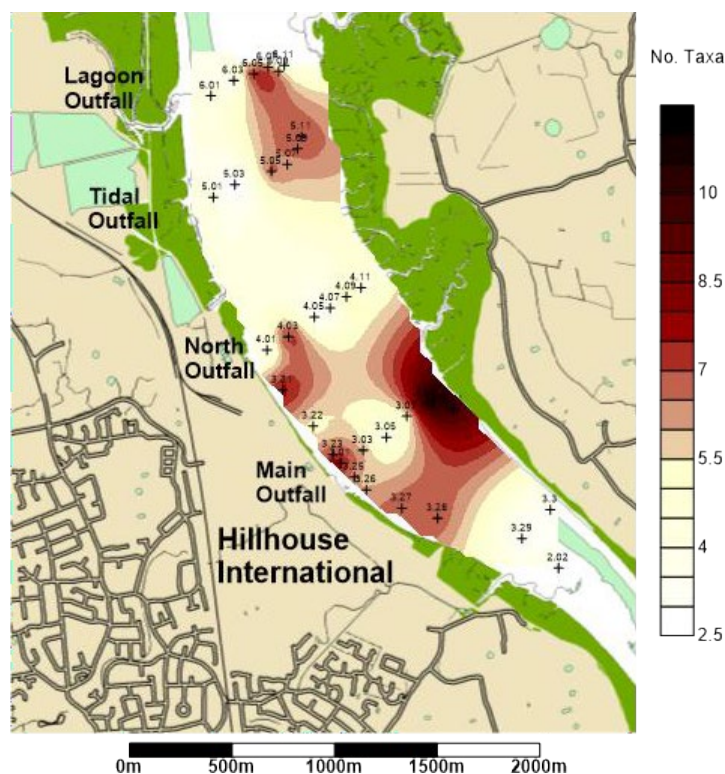
continues to be sediment granulometry. This is illustrated for the mean number of individuals in Fig. 7.

Fig. 7. Histogram showing the influence of the sediment type on the mean number of individuals (+/- SE) present at each station replicate.



By grouping the stations according to their predominant sediment types, it is possible to control for this variable within each group. No evidence of any impact from the site's effluent was observed on the benthic fauna in 2020. This was shown both in the uni-variate statistical analysis of metrics such as the number of taxa at each station (Fig. 8.) and in the multi-variate analyses of the data. Multi-variate analysis included the use of techniques such as multi-dimensional scaling (MDS) Similarity of percentages (SIMPROF) and statistical techniques that link the community structure to the concentrations of contaminants.

Fig. 8. Contour plot of the mean number of taxa at each station: June 2020.



Multi-variate analysis of the data from 1996 onwards shows that varying numbers of the dominant taxa within the estuary are primarily responsible for the differences observed from year to year. However, it was also shown that there were no significant differences in the benthic macrofauna in the estuary as a whole from year to year. The time series data also shows that any potential limited impact at stations close to the outfall that occurs in some years does not have any observable effect on the benthic macrofauna of the estuary as a whole.

It was concluded that the changes in the saltmarshes that have been observed since 2005 are largely a result of natural processes. The exceptions to this are some dead areas that were apparent in 2005 at SM 5 and which have subsequently recovered and some physical damage that occurred in 2012 and is in the process of recovery. It is thought that the dead area may have been due to what appeared to be a land drain (not connected to either AGCCE or Victrex) that discharged directly onto the top of the marsh in this area and had carved a channel down the marsh. The physical damage was caused by the recreational use of the marsh by motorbikes as a race track. None of these changes are a result of any inputs from the Hillhouse industrial complex.

From the results of this survey, it can be concluded that the effluent discharge from the Hillhouse industrial complex is not having a significant effect on the SPA, MCZ, SSSI or other protected habitats or species within the Wyre estuary.

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