

Design and Optimisation of Ion-exchange Technology for Defluoridation of High-Fluoride Groundwater in Tanzania – Theo Gill

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Research Aims

The project I was working on had three distinct objectives: first, I was to use graphical modelling to try and show how the fluoride adsorption capacity of the resin changed with varying levels of fluoride ions in the water being filtered, as well as investigating how successful computational models are at learning patterns in experimental data to predict adsorption data. I was also going to work in the lab trying to optimise the desorption of fluoride ions from the resin. Previous research had already identified the IDA-Al complex as the best performing resin for the filtration, but my aim was to find the eluent that best removes the fluoride ions from the resin once it has been used as a filter, thereby allowing the resin to be regenerated. Finally, I was involved in the development of a Techno-Economic Analysis of the filtration method, in order to prove its fiscal viability to shareholders and in the application for a competition from which we could receive funding to implement the filtration technique on a small scale.

Methodology

I started my research period with the modelling side of my work. I was provided with adsorption data of the resin adsorbing the fluoride ions in a solution of NaF, which included the concentration of fluoride ions in the initial solution and then the concentration of fluoride ions once the system had reached a state of dynamic equilibrium. In this state, fluoride ions are being adsorbed by the metal complex at the same rate at which the complex is desorbing other fluoride ions, and therefore the concentration of fluoride ions in the water remains constant. In practice, we took samples of the groundwater every 5 minutes using an automated liquid fraction collector and took the equilibrium concentration of fluoride ions to be the concentration at which the samples started to plateau. I then used C_e , the equilibrium concentration (mg/L), C_0 , the initial concentration (mg/L), V , the total volume of the solution (L), m , the dry mass of the absorbent added (g) and the following equation to calculate a value of q_e , which is the equilibrium adsorption capacity.

$$q_e = \frac{(C_0 - C_e) \times V}{m}$$

This value describes the amount of adsorbate, the fluoride ions, that is attached to the adsorbent, the metal complex, once thermodynamic equilibrium has been reached. Using the equations for the different adsorption isotherm models (Langmuir model, Freundlich model, D-R model, Temkin model, Redlich Peterson model and the BET model), I then tried to generate a similar set of data by adjusting the constraints of each equation until the calculated data sets matched the real data set as close as possible. This was achieved using the Solver Developer Tool on Excel, that would automatically adjust the calculator parameters in each equation to find the parameters that gave a data set as similar as possible to the real data set. I was also involved in testing different artificial intelligence models and seeing how successfully they could generate adsorption isotherms, and investigate the viability of relying on them completely to match different theoretical models to actual raw data. Although specialised machine-learning models that are trained specifically to handle scientific data, such as Artificial Neural Networks (ANNs) or Adaptive neuro-fuzzy interference systems (ANFIS), have been successful in refining research techniques by reducing the amount of raw data that has to be collected, I was trying to test how well more generic artificial intelligence systems could cope with identifying mathematical relationships between variables. I gave the initial and equilibrium concentrations of the fluoride ions in the solution to three different artificial intelligence models, Gemini 3.6 Flash, ChatGPT-5.6 Luna and Claude Sonnet 5. I then asked them to generate an adsorption isotherm for each of the adsorption models and match it as closely as possible to the raw data, as well as give me the values of the constants in the equations of each model that resulted in that closest match.

Once I had completed modelling the different isotherms, I started performing deloading experiments, where I used different eluents to try and identify the best performing one for fluoride capture. To gather the data, I first had to saturate the resin with fluoride ions, which I would do by filtering groundwater through the resin until an equilibrium concentration had been reached. For this first step, I had to set up a steady flow of lab-made groundwater, that was a close imitation of the groundwater in the regions where we want to implement the use of this filtration system, through the resin and prepare the automated liquid fraction collector to allow it to take samples of the filtered water at fixed intervals. To create the filter, I would remove the plunger from a syringe, place a piece of filter paper at the bottom of the syringe and then pack the resin on top, leaving about a centimetre of empty space in the syringe. I would then place one more piece of filter paper on top of the resin and place some small baking beads on top of it all to fully compress the resin. I would then attach a pump to each end of the syringe. The pump attached to the top would be attached to the groundwater, while the pump attached to the bottom would be dispensing the water into the test tubes in fraction collector. It was important to set the second pump at a higher rate than the first pump, otherwise the water would end up filling the syringe and spilling out from the top. I would set the fraction collector to rotate every 5 minutes, which would allow ample time for each of test tubes to fill with enough water to be analysed. Once the concentration of fluoride ions in the samples levelled off, I would take my eluent of choice, one of oxalate, bicarbonate, 0.1 mol NaOH, or NaCl

brine, and then set the system up so the elute was passing through the resin and being dispensed into empty test tubes in the fraction collector.



FIGURE 1. SYRINGE FILLED WITH RESIN

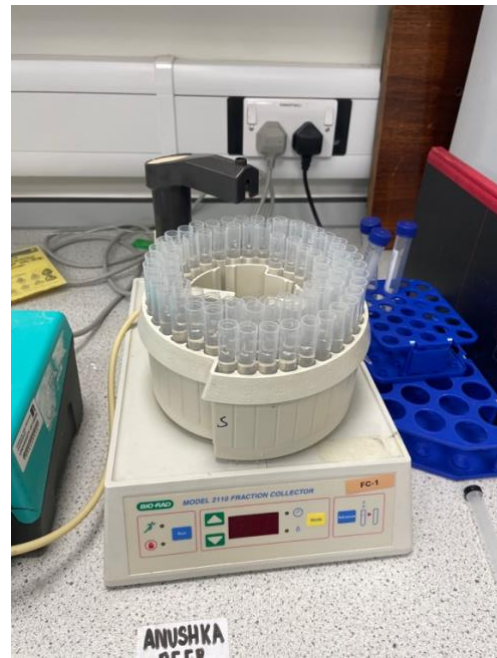


FIGURE 2. MODEL 2210 FRACTION COLLECTOR

I then had to analyse the samples by using an ion-selective electrode. This measured the concentration of each sample by measuring the potential difference through the sample, which is proportional to the concentration of the selected ion, the fluoride ions, in the sample. The first step was to calibrate the electrode using solutions of known concentration, measuring their concentration using the electrode and then drawing a calibration curve. I diluted solutions containing 10,000, 1,000 and 100 mg/L of fluoride ions down and mixed them with a Total Ionic Strength Adjustment Buffer (TISAB) to prepare them for analysis. I had to use TISAB for multiple reasons: first, it is a buffer solution that increases the concentration of ions in the solution being analysed. This will increase the potential difference across the sample, which will prevent any small background fluctuations in the voltage from affecting the results too much. TISAB also contains many chelating agents, such as CDTA, that bind to metal ions that could interfere with the results, of which there were many in our samples of groundwater. Once I had a complete calibration curve, I took every second sample from the fraction collector and prepared it for analysis, which meant mixing 2 ml of the fraction with 2ml of TISAB. Since the fraction collector filled up 80 test tubes, taking every second one still gave us a sufficient idea of how the resin would behave with different

eluents. Finally, I then had to generate graphs displaying clearly how much eluent was required for each regeneration experiment and calculate the mass of fluoride ions recovered by each eluent.

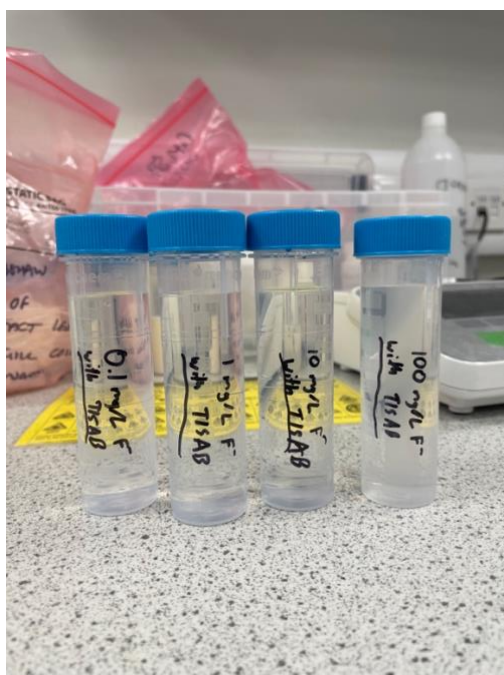


FIGURE 3. SAMPLES OF KNOWN CONCENTRATION USED TO GENERATE CALIBRATION CURVE

I also worked on experiments trying to show how scaling up the filter by increasing the amount of IDA-Al resin in the syringe would affect the effectiveness of the filter. These involved a similar process to the previous measurements but were more focused on the initial phase of the experiment, namely the loading of the resin. I would use the fraction collector and then record the concentration of the samples in the same manner as before, and then I compared how many samples it took for the water coming out of each filter to exceed the WHO's recommended maximum concentration of 1.5 mg of fluoride ions per litre of water.

When performing the Techno-Economic Analysis, I had to prove to potential shareholders that our filtration system was not only a feasible alternative to the biochar filtration systems in place currently, but rather superior by such a degree that it would prompt a replacing of the current system and its infrastructure. First of all, I had to think about the actual application of the filtration system, by considering how much clean water one system could generate before requiring regeneration, and therefore how many people it could provide for considering the average daily water consumption of one person. I then had to break down the cost of the system by considering all the components as well as any installation costs or training required to teach locals how to use the system. Furthermore, I also had to evaluate what effect the regeneration period would have on the overall costs: there would have to be an alternative water source, ideally a second filter using the resin, and the eluent being used to regenerate the column would have a cost as well, which was why it was important to identify the most efficient eluent.

Finally, I was also involved in preparing our team's application for a competition that would provide us with funding to pay for the installation of a prototype water filter. I had to collate the data we had gathered and identify what aspects of it were most important for the submission. A key challenge we had to overcome was presenting our data in a manner that could be understood by an audience

from a broad scientific background, and so the presentation required a lot of adjusting to produce a clear but still specialised entry.

Results

Having modelled the isotherms, I was able to produce a graph that displayed each isotherm and the data we measured. The experimental data shows a maximum fluoride adsorption capacity of $\approx 4 \text{ mg F}^-/\text{g}$ of absorbent, which is significantly higher than the maximum fluoride adsorption capacity for biochar equivalents. This graph can now be used to show to potential shareholders how the resin will perform at different concentrations of fluoride ions in the water that is being filtered.

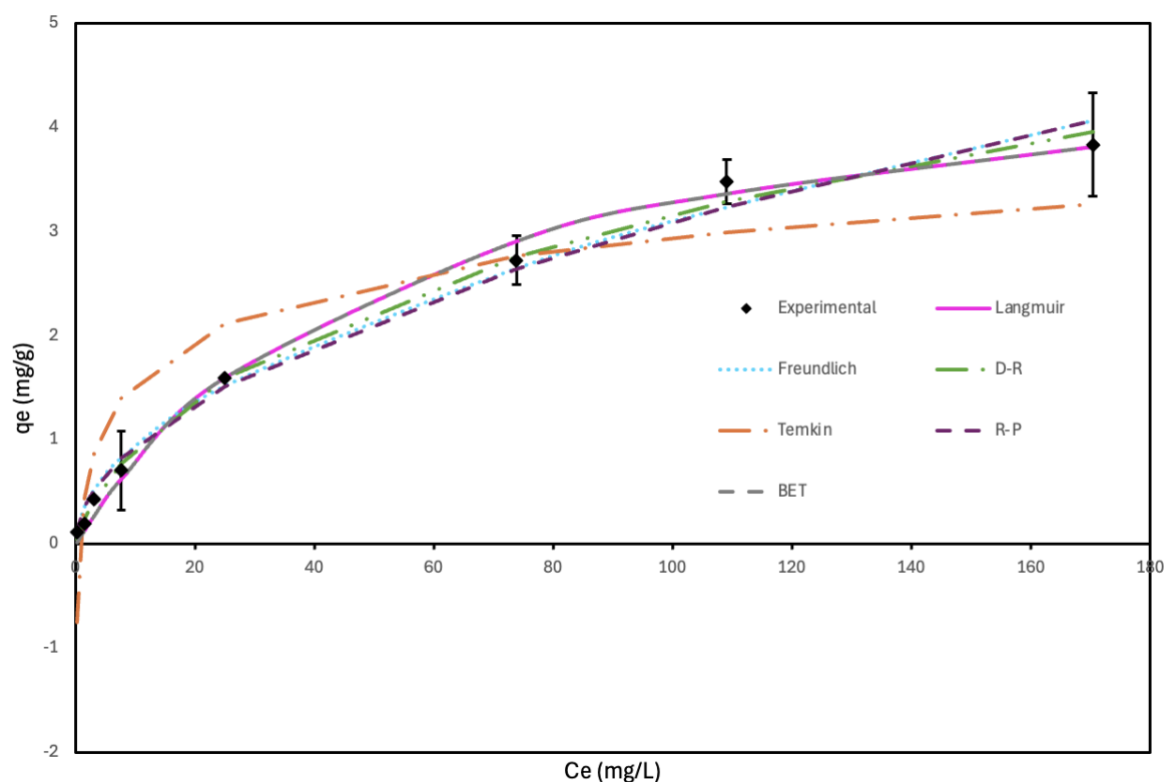


FIGURE 4. ISOTHERM MODEL DISPLAYING HOW DIFFERENT THEORETICAL ADSORPTION PROCESSES MATCH REAL DATA

My attempts to recreate such graphs using artificial intelligence were fairly successful: they were all able to match the majority of the adsorption models to the raw data I had provided them with to varying levels of success, but all three models struggled with at least one of the adsorption models, namely the Dubinin-Radushkevich model. This was likely due to the equation's reliance on the Polanyi potential, ϵ , which includes a logarithmic term. Because logarithms are extremely sensitive to minor fluctuations in low concentration values, the numerical solvers within the machine learning models likely struggled to accurately fit the raw data.

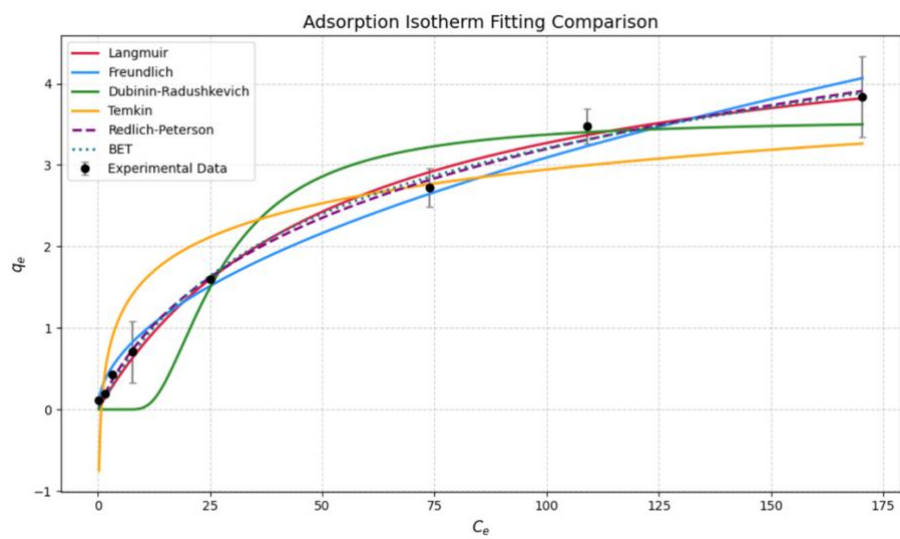
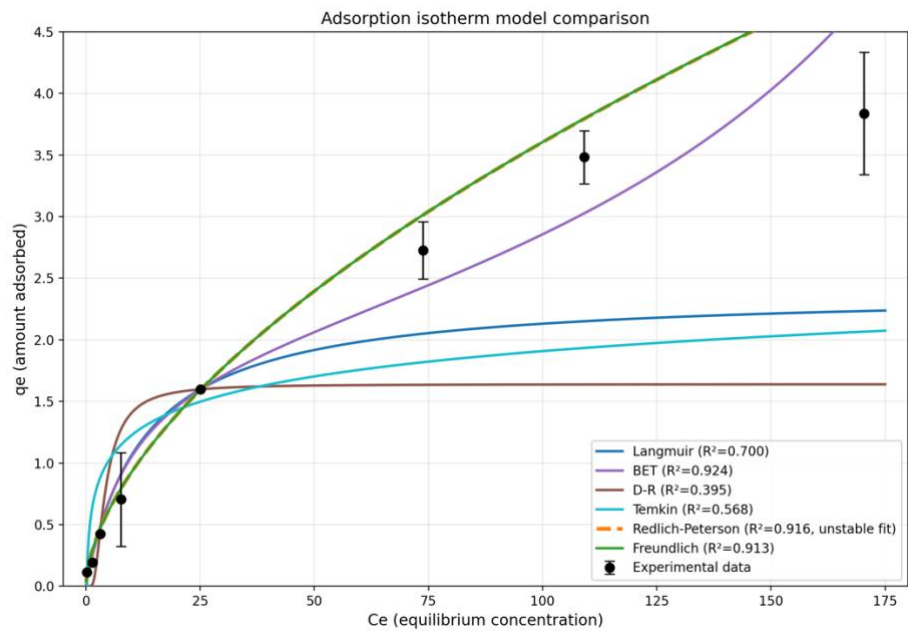
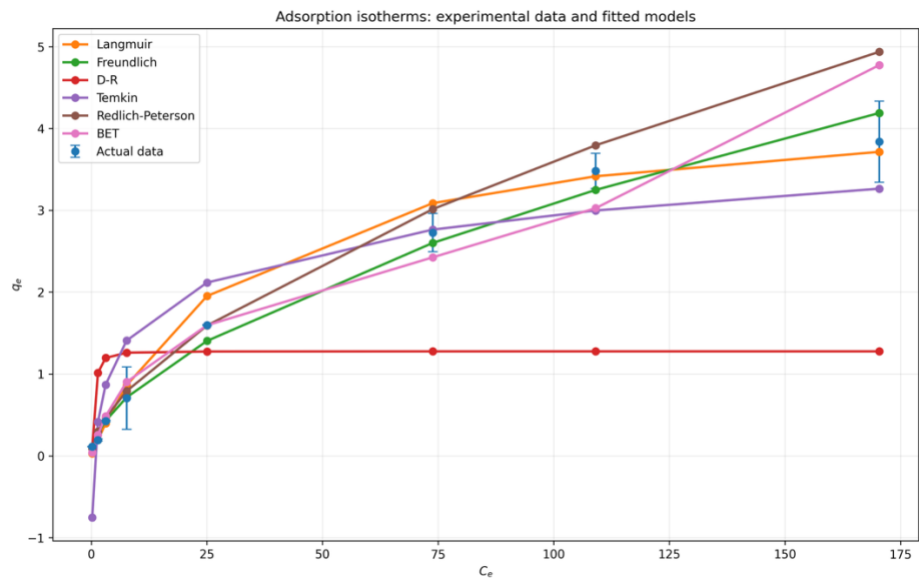


FIGURE 5. ADSORPTION ISOTHERMS GENERATED BY DIFFERENT AI MODELS (TOP – CHAT GPT-5.6 LUNA, MIDDLE – CLAUDE SONNET 5, BOTTOM – GEMINI 3.6 FLASH)

I also created graphs demonstrating the different efficacies of each eluent we used, to try and compare them and see which one would be most useful for recapturing the fluoride ions to regenerate the resin. Oxalate achieved the highest fluoride recovery by quite some margin, and so emerged as the best option to use.

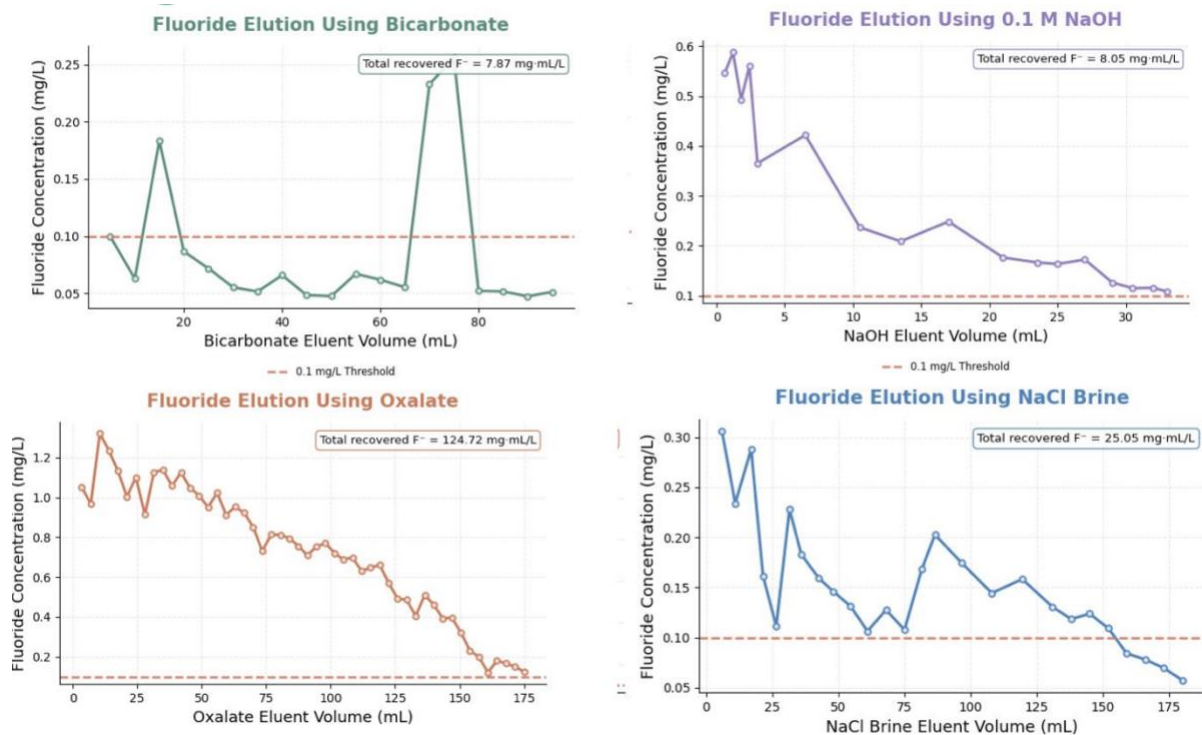


FIGURE 6. COMPARISON OF DIFFERENT ELUENTS USED FOR REGENERATION

Finally, I was also able to compare the effectiveness of two different sized filters using graphs to show clearly the advantages of using a larger filtering system: as shown below, the large-scale column is able to filter a much larger quantity of water to the WHO mandated levels, and so would require fewer regeneration cycles once in operation.

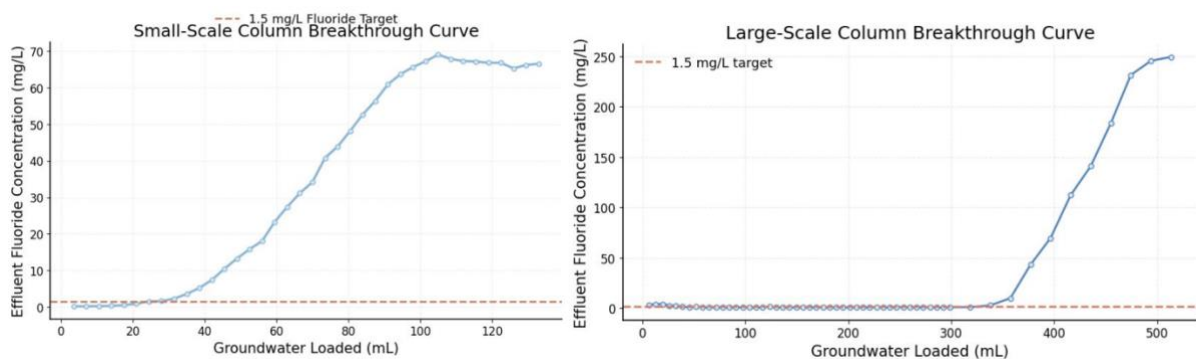


FIGURE 7. COMPARISON OF DIFFERENT SIZED FILTERS

Conclusion

Having worked on this project, I have been involved in trying to perfect a safe alternative for filtering groundwater in volcanic regions, where fluoride concentrations in water reach dangerous levels. Through the use of isotherm models, we have shown that this method of water filtration has a higher maximum fluoride uptake capacity compared to the methods currently being used, as shown below in Figure 4, especially when the initial fluoride concentration is higher.

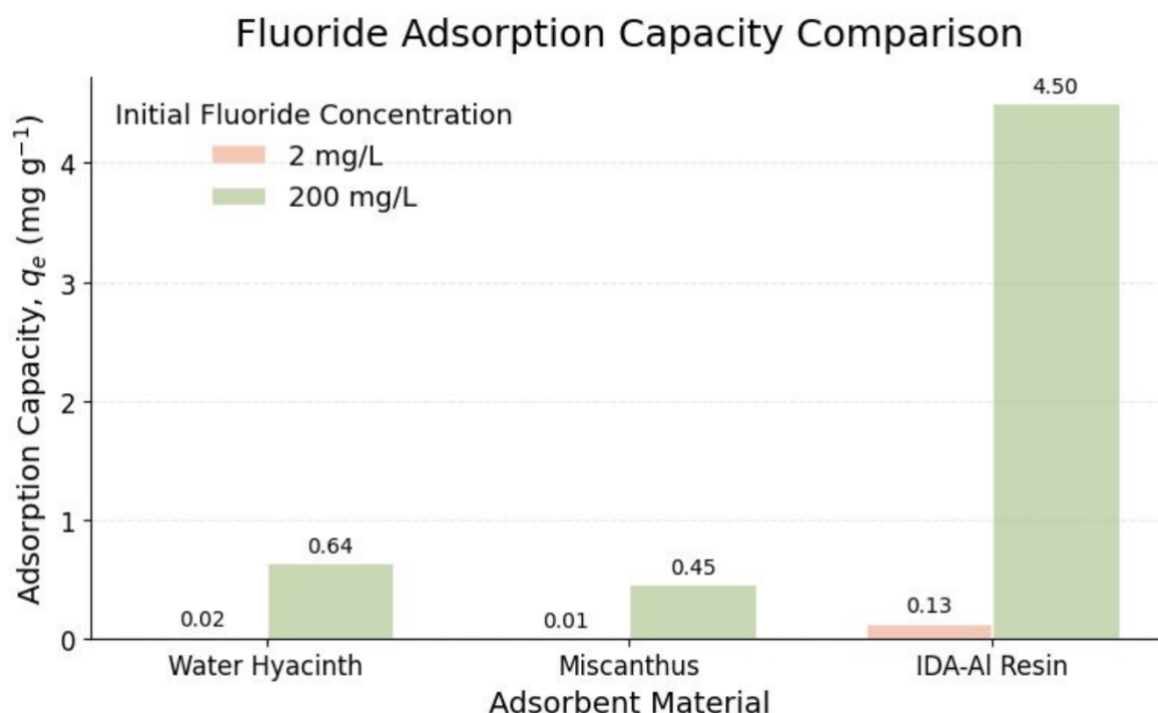


FIGURE 8. BAR CHART DISPLAYING MAXIMUM FLUORIDE ADSORPTION CAPACITIES OF DIFFERENT WATER FILTERS

Moreover, we successfully identified the best-performing eluent to accelerate the regeneration process as well as demonstrating that scaling the filtration system to filter more water would not inhibit its ability to produce safe drinking water.

Overall, artificial intelligence systems proved largely successful at generating adsorption isotherms from raw data, with the main exception being the Dubinin–Radushkevich model due to numerical sensitivity. Moving forward, I would like to work on refining my prompts by requiring explicit unit conversions and encouraging non-linear optimisation methods, that could significantly improve fitting accuracy for complex models.

Reflections

My involvement in this project proved difficult at first, due to the technical nature of the research and the need to develop my understanding of adsorption chemistry further before I could help my supervisor. However, I found that working with the isotherm models for my first two weeks was the ideal introduction to the project: it provided me with a broad overview of what we were trying to achieve, while still forcing me to understand the details of adsorption science. Having to work through each isotherm model provided me with the opportunity to learn about all the different adsorption mechanisms, what situations each one occurs in and how this would affect the kinetics

of the adsorption process. I also learnt about the two main different types of adsorption, chemical adsorption and physical adsorption, and how the nature of the bond between the adsorbent and the adsorbate depends on which of the two occurs.

During my research period, a scholar from Duke was staying in Leeds and working on the same project. I found this was a perfect opportunity to develop my leadership and collaboration skills. By delegating effectively, we managed to divide the work we had to do by adapting our tasks to our strengths. While I worked mostly on creating isotherm models, which was what I was most familiar with, she spent more time performing loading and deloading experiments and gathering further experimental data that I could then use. However, this also provided us with the occasion to improve our communication skills, as we spent a considerable amount of time familiarising each other with the part of the research we were more acquainted with: I explained how to use the Excel software to optimise the calculator parameters in order to minimise the error between the theoretical and experimental data, while she showed me how to set up the elution experiments and how to analyse the samples these produced. This proved to be very useful, as when she left, I was able to continue the experimental aspect of the research independently and efficiently, without having to spend too much time trying to understand the process of loading and deloading the filter column.

I found that my involvement in this project also taught me a lot about prioritisation and time-management. This was my first time working on technology that has such concrete real-world applications, and as such there were aspects of this project that I had not encountered fully before: for instance, I had to think about how to effectively present my data in a clear fashion. Although this is something I have encountered before, having had to write lab reports and display results throughout the course of my degree, I have never been faced with the prospect of having to pitch my results to any involved parties in an attempt to convince them our filtration system is more favourable. This project was also interesting as it gave me the opportunity to work with scholars from partner universities: in this case, academics from the Nelson Mandela African Institution of Science and Technology (NM-AIST) were helping relay our data to people in Tanzania and trying to identify somewhere we could start putting our filtration technology to use.

A part of this project that challenged me was the sheer amount of data I had to handle during both parts of my research: when I was modelling the adsorption isotherms, I had to take columns of data and adjust them so they would be compatible with the equations of each adsorption model, and when I was carrying out experiments I had to record 40 data points for the loading of the resin and then another 40 for the elution process. Therefore, I had to be very careful to clearly label each set of measurements and classify them so that they were easily accessible for future use.

Further steps

Having essentially identified the optimal conditions for this filtration system, the next step is trying to find shareholders to provide funding that would allow us to implement this technology on a small scale to prove the system's viability to replace other alternatives. I would like to travel to Tanzania next summer with the aim of identifying areas that would benefit most from an improved water filtration system, and try and work with the academics from NM-AIST to set up a pilot model with a demonstration system to hopefully provide the locals with enough training for them to be able to independently regenerate the resin when required and have their own self-sufficient water source. I would also like to help publish a paper to communicate our findings about the use of a metal complex as a regeneratable water filter to a wider scientific audience.