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Response Surface Methodology for the Production of a Filter Membrane from the Combination of Sand, PET and Salt as a Pore-Forming Agent: Application to the Filtration of Wastewater from Septic Tanks

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Abstract---Plastic waste is increasing every day due to urbanization, population growth and in turn, pollutes the environment. These wastes are considered to be a big problem due to their very low biodegradability and presence in large quantities. A large amount of plastic which is being brought by human activities is discarded or burned which leads to the contamination of the environment and air. Therefore, finding alternative methods of disposing of waste by using kind approaches is becoming a major research issue. In this research, polyethene terephthalate waste is used as a binder by burning and mixing with sand and sodium carbonate to investigate the possibility of producing composite material with a plastic sand filter and study the effect of sand, polyethene terephthalate waste and porogen with different design mix ratio on the properties of the product. An experimental design plan was adopted to formulate the materials. To fabricate this material, the melt moulding/particulate leaching technique was used, in which the components are mixed in their respective proportions, moulded, melted and afterwards leached to remove the porogen. We obtained an optimum of 31.0711% porosity for the optimum mixture containing proportions of 0.5, 0.25 and 0.25 plastic sand and porogen respectively. An improved melt/moulding particulate leaching technique was used to improve the permeability and reduce pore sizes of the optimum. This technique involved using a saturated solution of porogen instead of using the porogen in particulate form. The microbial cut-off efficacy of the material was evaluated, in which microbiological analyses were used to enumerate the number of microbes in a lake water

sample before and after filtration through the porous material. The results of viable cell counting showed that the material eliminated 92.6% of the water microbes analysed.

Keywords---composite material, experimental design plan, plastic waste, polyethene terephthalate, porogen, properties.

Introduction

Accelerated plastic use over the years has created an enormous quantity of waste plastic. In 2021, 390.7 million metric tons of waste plastic was produced globally. Despite the continued increase in plastic production, only 9% of this volume is recycled. For the plastic that does not get recycled, 22% is mismanaged, 19% is incinerated, and the remaining 50% is directed to landfill. This suboptimal waste plastic disposal has widespread negative environmental effects. The vast amount of unrecycled plastic presents a substantial opportunity to profitably utilize available materials, reduce pollution, and redirect waste from landfills. To take advantage of that opportunity, researchers have investigated methods to use waste plastic in new products. One such application of waste plastic that continues to be investigated is the use of polymers as a replacement aggregate or fiber reinforcement in concrete. Traditional concrete is the most used material globally, with approximately 30 billion metric tons of concrete being consumed each year. The manufacturing of concrete, however, also makes it one of the most detrimental materials to the environment. A typical concrete mixture consists of 12% Portland cement, 34% sand, 28% crushed stone, and 6% water by weight. To reduce the total emissions from concrete, many studies have been conducted in search of a less energy-intensive binder to replace Portland cement. These existing studies have investigated the use of industrial waste products as supplementary cement materials (SCMs) and have already demonstrated some select successful replacements for Portland cement. The problem with concrete, however, is more severe than plastic, with the total weight of plastic produced in sixty years being matched by concrete in only two. Beyond carbon emissions, concrete is also responsible for demanding 18% of global industrial water consumption and 9% of global industrial water withdrawal annually (Ellis et al., 2020; Woods, 2023). In this regard, terms such as green materials, sustainable development, and eco-efficiency construction are becoming more extensively acknowledged and used. Zhao et al. (2020); and Majhi & Nayak, (2020), stated that the consumption of waste and recycled materials has become an alternative solution to reduce the utilization of raw materials in construction industries. This usage of waste materials could significantly help reduce disposal and landfilling spaces, decrease environmental pollution, and lower the cost of construction. Accordingly, green production awareness inspires the construction industries by using waste materials such as aggregates, cementing materials, and fibrous materials instead of natural resources. In Cameroon, as in many countries of the world, the amount of plastic is increasing and occupies a large part of solid waste. It is used in automotive, electronics, buildings and other industries. However, plastic also has negative consequences, such as emissions of greenhouse gases or ecological damage. Usually, plastic is a non-biodegradable material, and therefore the waste remains for long periods in the environment, representing a risk to human health and the ecosystem (Alexandra, 2022). The volume of waste is only one problem. Recycling this type of waste to produce new materials like concrete or mortar appears as one of the best solutions, due to its economic and ecological advantages. Previous studies showed that it was possible to use various plastic waste as aggregates or fillers or fibers in concrete such as polystyrene (PS) (Tang et al., 2008), expanded polystyrene (Kan & Demirboğa, 2009), polyvinylchloride (pvc) (Kou et al., 2009), low-density polyethylene (LDPE) (Chaudhary et al., 2014), high density polyethylene (HDPE) (Naik et al. 1996; Mehdi Seghiri et al., 2017), E-plastic waste (Manjunath, 2016; Alexandra, 2022; Ge et al., 2013). The researchers indicated that the waste plastic could be reused as partial substitutes for sand (Aragh et al., 2015; Kou et al., 2009; Manjunath, 2016). The research revealed that the incorporation of any type or form or size of plastic waste as aggregates generally decreases the dry density (Tang et al., 2008; Manjunath, 2016; Ismail & Al-Hashmi, 2008; Kan & Demirboga, 2009; Choi et al., 2005; Akçaozöğlü & Akçaozöğlü, 2010; Ge et al, 2013). The improper disposal of post-consumer PET raises environmental issues because PET is not biodegradable (Frigione, 2010). Also, the gases produced by incinerating PET cause air pollution and public health concerns. Thus, one of the logical methods to address PET waste is to recycle it for industrial use (Akçaozöğlü & Akçaozöğlü, 2010; Ge et al., 2013). This project, therefore, aims to develop a new generation of cheaper, green porous materials, by studying the use of a new resource: plastic waste. This fundamental change in research perspective can, in combination with existing research efforts, lead to cheaper and more environmentally sustainable porous materials technology. These two factors are essential in incentivizing industry to implement porous materials technology. The specific objectives include the formulation, fabrication characterization and performance evaluation of the porous

composite material from plastic waste; and the design of a soak pit to investigate the possibility of scaling up this technology.

Raw Materials

Plastic wastes

Plastic waste used is mainly from beverage bottles with a basis of polyethene terephthalate.

Table 1
Physical properties of PET (RESEDA, 2006)

Density	1.35g/cm ³
Absorption of water	0.001%
Elastic modulus	2.76-4.14
Melting point	265°C

Sand and salt

The sand used here was silica rolled granular (grains of sizes 500µm-1mm). This was sodium chloride (NaCl), a normal food-grade table salt. This porogen was chosen because it is very soluble in water, cheap and readily available. The oven was of mark “fours nagat”, used for melting the mixture. It was capable of heating at a maximum of 950°C at a rate of 10 °C/min, with a power rating of 6300W.

Grinder: this is composed of a polisher mark METASERV universal polisher, adapted to grate by fitting a grater to its turntable. It has a maximum speed of rotation of 400rpm. The press used here was a hand screw press and the Mould: was disc-shaped steel with a height of 7mm and a diameter of 30mm. Statgraphics Centurion 16.1.11 (used to perform statistical analyses, create and analyse experimental designs), Solidworks 2013 for drawing.

Methods collections of raw materials

Plastic PET bottles were collected through the municipal waste of Ngaoundere town and Sand was obtained from the river. The porogen was table salt.

Preparation of raw materials

➤ Obtention of PET powder:

PET bottles were grated (shredded) to obtain plastic shreds. This was done by using a grater fitted to the turn-table of a polishing machine (METASERV, universal polisher) rotating at a maximum speed of 400rpm. These PET shreds were then sieved to obtain PET powder with particles of diameters, $d \leq 500\mu\text{m}$.

➤ Experimental design for the process

The mixture design class comprising a simplex centroid design was used to model three factors; the proportion of each component (plastic, sand and salt). The software STATGRAPHICS Centurion 16.1.11 was used to generate the experimental design. The experimental domains over which factors were studied were based on tests and literature. These independent variables were studied using the mixture design methodology at two different levels (-1, +1) to give a total of 14 experimental runs.

Mixture design

Experimental factors include:

➤ Proportion in plastic(X_1), Proportion in sand(X_2), Proportion in porogen (X_3)

The responses here are: porosity(Y)

Table 2
Domaine of factors

Independent variable	Code levels	
	-1	+1
Proportion of plastic	0.5	0.65
Proportion of sand	0.25	0.4
Proportion of salt	0.1	0.25

4.4 Fabrication`

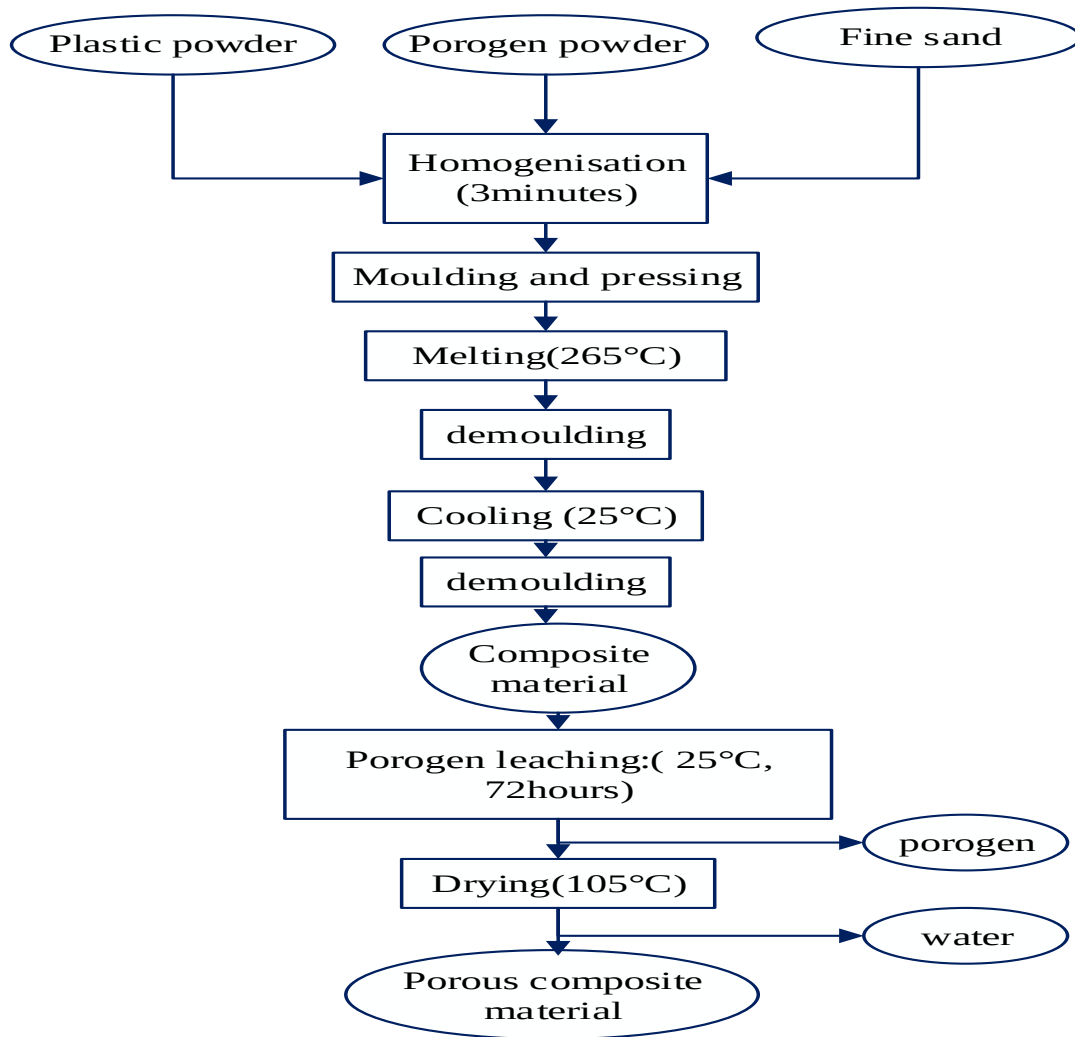


Figure 1. Process flow diagram for the melt moulding porogen leaching technique

Production of the PET PET-based porous Composite by the melt Moulding/Porogen leaching technique. Filters were produced by a combination of melt moulding and particulate leaching techniques. This method was preferred over other methods because of its relative simplicity; versatility; Ease of controlling pore size and geometry.

Characterization of material

Porogen Leaching Test:

This test was carried out to ascertain the complete removal of the porogen and to determine the minimum and maximum time needed to completely leach out the salt (NaCl). The porogen leaching was performed at room temperature in beakers containing 15 mL of distilled water. After every 4 hours, 10mL of leachate was withdrawn and titrated by silver nitrate solution to check the percentage of porogen leached out from the material. This titration was done using the Mohr's method for the titration of chlorides. The leaching process was performed only on the material having the greatest proportion of porogen (0.25).

Porosity of the composite: the total saturated water method

Porosity ϵ is the fraction of the total material volume that is taken up by the pore space. Thus it is a single-value quantification of the amount of space available to fluid within the body of material. Being simply a fraction of total volume, ϵ can range between 0 and 1, or 0 and 100%.

The porosity of the material was calculated by measuring the volume (V_s) and volume of the pore (V_p) of the sand/plastic composite material. V_s was calculated using the geometry of the material i.e. length, width and height, using the total saturated water method. The weighted material (M_0) was immersed in water at room temperature. The system was maintained for 15 min in desiccators under reduced pressure to remove air bubbles. The materials were taken out and wiped superficially with a filter paper to remove the surface water, and then weighed immediately (M_1). V_p was defined as: $V_p = (M_1 - M_0)/\rho_w$ (2) where ρ_w (1g/ml) represents the water. The porosity of the material was then calculated according to the following equation: ϵ (in %) = $(V_p/V_s) \times 100$ (3)

Performance evaluation of optimum

Evaluation of permeability

The permeability of a porous material is an important performance indicator of the throughput or flux. This therefore gives information on the extent of pore interconnectivity. This is typically expressed by the pure water permeability (PWP), which is a measure of the pure water flux through the material per unit of pressure difference:

The material permeability is tested in a dead-end filtration setup. In this module, the feed solution is in contact with the material surface (effective surface area of 7.065cm²), at normal atmospheric pressure.

The module is filled with distilled water and the pure water permeability is calculated using the following equation: $PWD = (V_p / A_m \cdot t \cdot P)$ (4)

Where V_p is the permeate volume in litres (L), A_m is the membrane surface area (m²), the permeation time in hours (h), and P is the pressure (bar). This test was carried out on three samples of the two



Figure 2: filter model optimum (having component proportions: 0.5 plastic, 0.25 sand, 0.25 porogen) fabricated by the two different methods: the melt moulding and the improved melt moulding porogen leaching technique

Microbiological Analyses

The purpose of these analyses was to evaluate the performance of the porous material in eliminating water microorganisms. Therefore, to do this, these analyses were carried out by detection and enumeration of viable microorganism cells present in a water sample before and after filtration through the porous material.

Enumeration of microorganisms: here, the microbiological analyses permit the quantification of a particular group of microbes present in the sample analyzed. The results of the enumeration analyses are reported in the concentration of the particular type of microorganisms per unit volume of sample analyzed. A viable cell count allows one to identify the number of actively growing/dividing cells in a sample. The plate count method or spread plate relies on bacteria growing a colony on a nutrient medium. The colony becomes visible to the naked eye and the number of colonies on a plate can be counted.

Results and Discussions

Formulation

The following table shows the proportions of each component in each of the 14 experimental runs.

Table 3
Proportions of components in formulation

Code	Proportion In Plastic	Proportion In Sand	Proportion In Porogen
M1	0.65	0.25	0.1
M2	0.6	0.3	0.1
M3	0.6	0.25	0.15
M4	0.55	0.35	0.1
M5	0.55	0.3	0.15
M6	0.55	0.25	0.2
M7	0.5	0.4	0.1
M8	0.5	0.35	0.15
M9	0.5	0.3	0.2
M10	0.5	0.25	0.25
M11	0.55	0.3	0.15
M12	0.6	0.275	0.125
M13	0.525	0.35	0.125
M14	0.525	0.275	0.2

Fabrication



Figure 3. Photos of some samples of porous material fabricated

These samples show no clear distinction in colour because they were heated at the same temperature within the same amount of time.

Porogen leaching test

Figure 4 shows the variation in the percentage of porogen leached out with time.

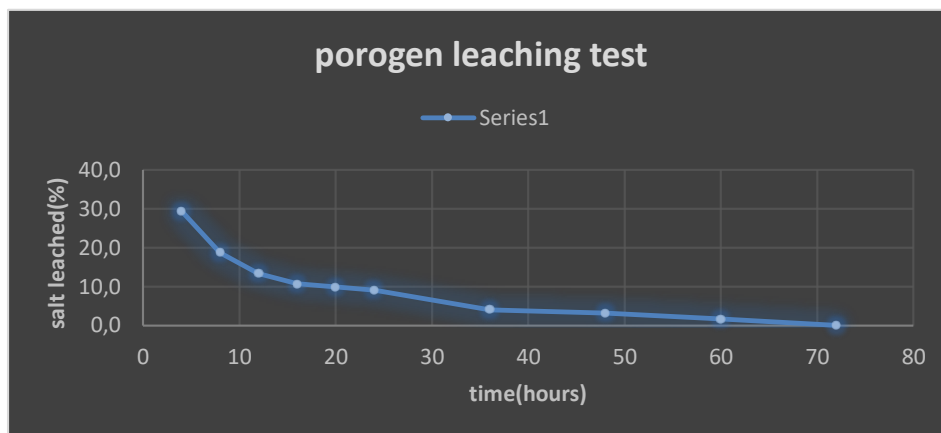


Figure 4. Porogen leaching Test (at room temperature)

Ideally, this graph should have been a straight line. But this is not the case here. This deviation can be explained by considering the material as composed of cores. The cores resulted from the pressing step in the fabrication of the material. It is assumed that the innermost core is the hardest. The Harder the core the more tightly the held are the porogen particles in the matrix. Titration was done every 4 hours. Within this time, the assembly containing the material immersed in the distilled water was agitated periodically. The graph therefore shows that it takes time for the distilled water to seep into the pores and dissolve the porogen. This dissolved porogen only comes out as the leachate upon agitation. 75% of the initial porogen was leached out within the first 24 hours corresponding to the porogen at the surfaces and in the inner core of the material. The remaining 24.3% was left trapped in the innermost core of the material took a longer time to be leached out.

Characterisation of the porous materials

Influences of the component's proportions on porosity

Table 4
Effects of component proportion on porosity

Code	Proportion of plastic	Proportion of sand	Proportion of porogen	Mdry (g)	Mwet (g)	Vp (cm ³)	Vs (cm ³)	Porosity (%)
M1	0.65	0.25	0.1	14.42	14.8	0.38	3.462	10.99
M2	0.6	0.3	0.1	11.95	12.39	0.44	3.674	12
M3	0.6	0.25	0.15	12.59	13.11	0.52	3.603	14.3
M4	0.55	0.35	0.1	12.11	12.65	0.54	3.603	15.11
M5	0.55	0.3	0.15	13.01	13.79	0.78	4.098	19
M6	0.55	0.25	0.2	12.09	13.06	0.97	3.886	24.86
M7	0.5	0.4	0.1	12.17	13.03	0.86	3.744	23
M8	0.5	0.35	0.15	13.45	14.29	0.84	3.674	22.87
M9	0.5	0.3	0.2	12.08	13.01	0.93	3.603	25.9
M10	0.5	0.25	0.25	12.42	13.61	1.19	3.815	31.1
M11	0.55	0.3	0.15	12.44	13.1	0.66	3.462	19.2
M12	0.6	0.275	0.125	12	12.48	0.48	3.603	13.44
M13	0.525	0.35	0.125	13.1	13.74	0.64	3.533	18.23
M14	0.525	0.275	0.2	12.79	13.72	0.93	3.674	25.4

Vs is the volume of solid calculated from the geometry of the solid and Vp is the volume occupied by pores

This table can be better understood by presenting the variation of the porosity as a function of the component proportion in a graphical form Figure 4.

Variation of porosity with component proportion

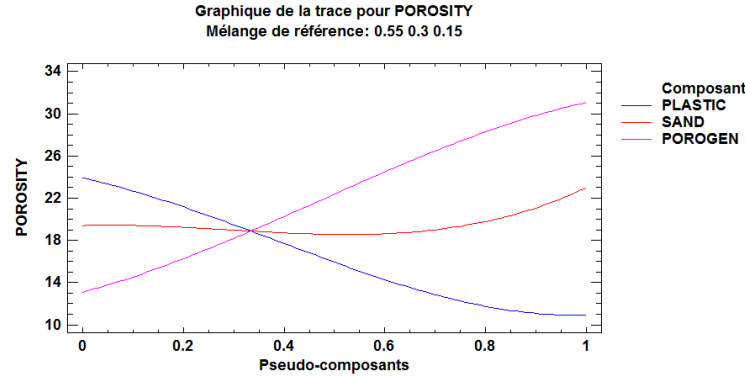


Figure 5. Variation of porosity with component proportion

The slopes of the graphs show that the porosity of the material is affected by all components, with the sand and the porogen having a positive effect, the latter having a much greater effect on the porosity. Plastic proportion influences the porosity negatively.

The graph of sand proportion against porosity shows three distinct zones of variation:

Between 0 and 0.35: the graph of variation of sand proportion against porosity stays almost constant. This means that at very low sand proportions, the porosity of the material is largely due to the porogen and thus, the sand does not improve the porosity within this range.

Between 0.35 and 0.55: the graph slightly slopes downward, affecting porosity negatively.

Between 0.55 and above: the graph steeply slopes upward, meaning a large effect on porosity due to variation of the sand proportion.

These observations can be better explained by considering the sand/plastic ratio. Here, we consider the mixture as a particle-reinforced composite in which the sand particles have been dispersed in a plastic matrix. In this light, the porosity would be affected by the strength of binding between the sand particles and plastic (binder) surrounding them. The binding strength reflects how tightly held the sand particles are to the plastic. If this binding strength is weak the sand particles are very loosely held by the plastic, meaning that there will exist some gaps (voids) at the sand/plastic interphase (boundary), which accounts for porosity.

At low sand/plastic ratios (below 0.35), the plastic acting as the binder, the binder proportion is more than enough for the binding strength to be affected. Thus, porosity is the least affected. On the other hand, at very high sand-plastic ratios (above 0.55), the binder proportion is not enough to sufficiently and tightly bind the particles leading to a large decrease in the binding strength. This large decrease in the binding strength leads to a large increase in porosity.

However, there is a particular sand/plastic (0.45 to 0.55) ratio at which the binding strength is maximum. The sand/plastic interphase is so tightly closed that there are no voids or gaps between them. This is shown by the relatively small decrease in porosity.

Modelling and statistical analyses of porosity

➤ Significance of model

Table 6
Estimated full model effects for porosity

Source	Sum of squares	Df	Mean square	F-ratio	p-value
mean	5417.51	1	5417.51		
linear	448.695	2	224.348	90.67	0.0000
Quadratic	16.3905	3	5.4635	4.04	0.0508

Source	Sum of squares	Df	Mean square	F-ratio	p-value
Special-cubic	1.19236	1	1.19236	0.87	0.3830
Cubic	8.66977	3	2.88992	11.97	0.0182
Error	0.965874	4	0.241469		
Total	5893.43	14			

These results show that only the linear and the cubic models are statistically significant as shown by the p-value.

➤ Validation of model

The results for the full model show the SE (square root of MSE), R-squared, and adjusted R-squared values.

Table 7
Full model results

Model	SE	R ²	Adjusted R ²
Linear	1.57303	94.28	93.24
Quadratic	1.1634	97.72	96.30
Special-cubic	1.17325	97.98	96.24
Cubic	0.491394	99.80	99.34

The SE is smaller and the adjusted R-squared value is larger for the Cubic model than it is for the Linear, Quadratic and special cubic models. These results indicate that the Cubic model is better than the linear model for estimating the porosity.

➤ Significance of components and terms

Table 8
Cubic model fitting results for porosity

Parameter	Estimate	Standard error	T-Statistic	Probability
A:PLASTIC	10.9263	0.490183		
B:SAND	22.9825	0.490183		
C:POROGEN	31.0711	0.490183		
AB	-15.44	2.19181	-7.04439	0.0021
AC	-6.37255	2.19181	-2.90743	0.0438
BC	-12.5001	2.19181	-5.70307	0.0047
ABC	27.8755	12.5443	2.22215	0.0904
AB(A-B)	9.87544	4.19904	2.35183	0.0784
AC(A-C)	-23.1382	4.19904	-5.51037	0.0053
BC(B-C)	-3.17869	4.19904	-0.757003	0.4912

All components and terms are statistically important ($p < 0.05$), except the ABC and AB (A-B) and the BC (B-C) whose p-values are greater than 0.05. Thus, a change in these components significantly affects porosity.

➤ Overall model significance

Table 9
ANOVA for porosity

Source	Sum of squares	Df	Mean square	F-ratio	Probability
Cubic model	474.948	9	52.772	218.55	0.0001
Total error	0.965874	4	0.241469		
Total (corr.)	475.914	13			

Optimization of response

Mathematical model: cubic

$$Y_1 = 10.9263 \cdot X_1 + 22.9825 \cdot X_2 + 31.0711 \cdot X_3 - 15.44 \cdot X_1 \cdot X_2 - 6.37255 \cdot X_1 \cdot X_3 - 12.5001 \cdot X_2 \cdot X_3 + 27.8755 \cdot X_1 \cdot X_2 \cdot X_3 + 9.87544 \cdot X_1 \cdot X_2 \cdot (X_1 - X_2) - 23.1382 \cdot X_1 \cdot X_3 \cdot (X_1 - X_3) - 3.17869 \cdot X_2 \cdot X_3 \cdot (X_2 - X_3)$$

Response surface for porosity

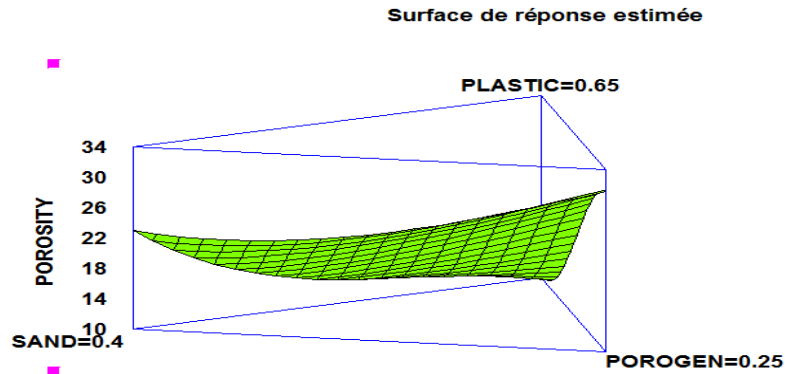


Figure 5. Response surface for porosity

Objective: maximize porosity

Optimum value: 31.0711

Factor	Low	High	Optimum
PLASTIC	0.5	0.65	0.5
SAND	0.25	0.4	0.25
POROGEN	0.1	0.25	0.25

Performance Analyses of Optimum

Permeability to water test (pwt)

The results shown in Table 10 are results obtained for the dead-end filtration through a 7.065cm² surface area of the porous material fabricated.

Table 10
Volume of filtrate for triplicate filtration trials over 24 hours at atmospheric pressure

	Nature of porogen used to fabricate material	
	particulate	Saturated solution
Average	308.3±12mL	216±9mL
pwp	436.38±11.2 L/m ² /day	305.73 ±16L/m ² /day

These results show that the sample in which the porogen was in particulate form is more permeable compared to when the porogen is introduced as a saturated solution. This is because the pore size ranges are not the same for both samples. Since filtration was done at atmospheric pressure, the sample with the smallest pore size range will have the smallest fluid flux at a given time interval.

Microbiological analyses

Viable cell counting: The number of viable cells was reported as the number of colony-forming units per milliliter (CFU/mL) of the original water sample. These results show that the lake water was heavily polluted with animal faeces as shown by the high number of faecal streptococci and faecal coliforms CFU/mL recorded.

Microbial cut-off efficacy of the porous material

The effectiveness of the material was calculated according to the MICROCHEM method, as the percentage of the reduction of the microbes present in the original water sample eliminated by filtration through the material (eqn9).

$$\% \text{reduction} = \left(1 - \frac{\text{CFU}_i}{\text{CFU}_f}\right) * 100 \quad (9)$$

CFU_i is the number of colony-forming units per milliliter of the initial water sample

CFU_f is the number of colony-forming units per milliliter of filtered water sample

Table 11
Effectiveness of material in eliminating common water microbes

Bacteria	CFU _i	CFU _f	Percent reduction (%)	Average percent reduction
Total flore	6.050*10 ³	950	84.3	92.6
Total coliforms	1.36*10 ⁴	380	97.2	
Faecal coliforms	2.09*10 ⁵	1180	99.4	
Faecal streptococci	3.950	415	89.5	

Design realization

Therefore, for a 5-member family, a circular soak pit of diameter 1m and a depth of 2.5m is required to safely dispose of the septic effluents. Thus, the corresponding area of the porous material necessary to infiltrate a daily sewage loading of 1400L is found to be 4.6m²

Figure 6. Technical drawing of filter media lined pit showing its dimensions

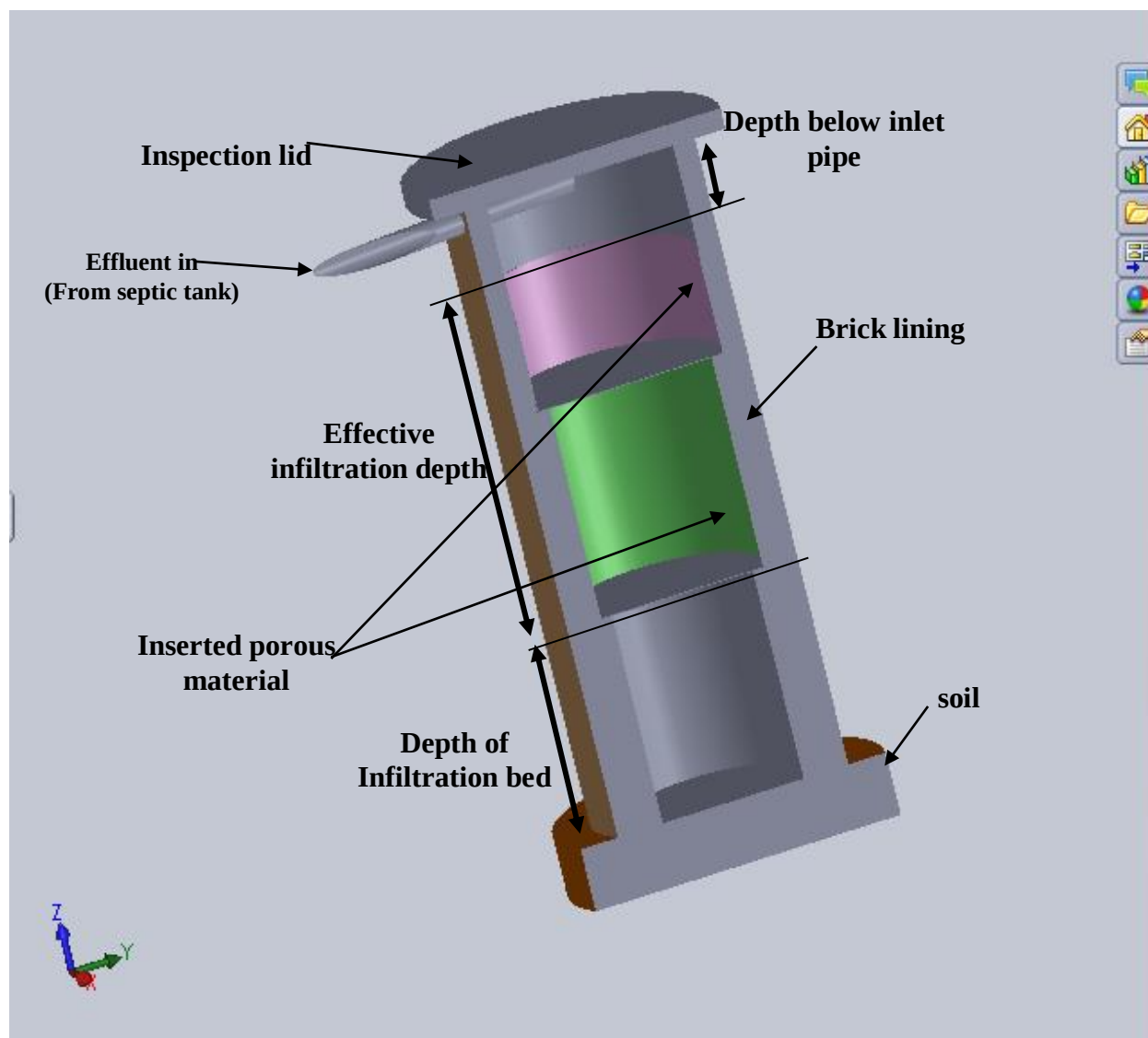


Figure 7. Section view of designed soak pit

Conclusion

The main objective of this work was to fabricate a green porous composite material for the elimination of water microorganisms in a cost-effective and environmentally friendly way. This was a contribution to the recycling of plastic waste. From the experimental design plan, all three components have a great influence on the porosity of the fabricated material, with the porogen and plastic proportions having the greater influence. The sand only had an effect at very high proportions. Thus, the objective was attained with a 92.6% elimination of the water microbes from the water sample analyzed. The optimization gave a maximum porosity of 31.2% with proportions of 0.5 plastic, 0.25 sand and 0.25 porogen. Plastic waste-derived porous materials are fundamentally possible and promising for application in septic soak pits for the elimination of bacteria from septic effluents. From future perspectives, we suggest that a proper mechanical characterization of the fabricated porous materials should be done. In this project, we have considered mechanical characterization as given in the literature for a sand/PET composite only. This therefore ignores the effect on the rigidity of the resulting material due to the porogen. Thus, a future characterization of the material rigidity and compressive strength should be done and a proper characterization of pore sizes be done to make sure that the porosity of the material is uniquely due to the porogen.

Conflicts of Interest: The authors declare no conflict of interest

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