

OPTIMIZATION OF TPS FILMS USING AN ADAPTIVE DESIGN OF EXPERIMENTS APPROACH IN A BAYESIAN OPTIMIZATION FRAMEWORK

Abstract

Plastic pollution, amounting to 12 million tons annually, necessitates sustainable alternatives to single-use plastics. Compostable thermoplastic starch (TPS) films show promise but lack strength and durability compared to traditional plastics. This study employs an adaptive design of experiments (DoE) approach to enhance TPS films by optimizing testing points. The research focuses on varying concentrations of plasticizers (acetic acid and glycerol) in a water and potato starch mixture, aiming to identify the optimal ratio maximizing tensile strength and % elongation at break. Gaussian process regression (GPR) with uncertainty estimation and Bayesian optimization (BO) utilizing an acquisition function (AF) are employed. The AFs are compared to determine the best-suited one, iteratively updating the model until minimal improvement is predicted. Results indicate TPS films with 1.06% plasticizer weight exhibit the highest tensile strength (2.33 MPa) and % elongation at break (93.45%). Further testing is recommended to validate GPR and BO models and explore additional factors like adjusting the potato starch to plasticizer ratio for enhanced TPS film properties.

Keywords

thermoplastic starch, Bayesian optimization, Gaussian process regression, tensile strength, elongation at break, adaptive design of experiments, machine learning

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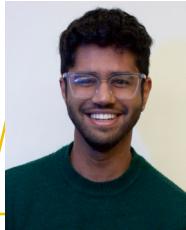
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INTRODUCTION

Fossil-based plastic bags are detrimental to the environment as they do not biodegrade into safe by-products. Instead, they break down into microplastics that pollute the marine and terrestrial ecosystems. Every year, five trillion fossil-based plastic bags are produced in the world, and less than 1% are recycled (The World Counts, 2023). When a plastic bag reaches the ocean, it takes up to 1,000 years to decompose despite being used for only 12 minutes (Bruhns, 2022). Humans are also affected by plastic pollution since when plastic breaks down into smaller particles, it turns up in the food we eat. Unless serious action is taken to mitigate this problem, it is estimated that the weight of ocean plastics will exceed the combined weight of all the fish in the seas by 2050 (Reddy, 2018).

This paper addresses the urgent need for environmentally friendly alternatives to petroleum-based plastic bags. This study focuses on improving these thermo-plastic starch (TPS) films to make them a more sustainable replacement not just for traditional bags, but also for current TPS films on the market today. This research aims to create a film that biodegrades into food-safe molecules that pose no threat to plant and animal life and are produced in a more environmentally conscious manufacturing process in which the film takes less energy to create than traditional plastics. Current TPS films lack the mechanical properties, primarily tensile strength and elongation at break, necessary to replace the typical petroleum-based plastics (Diyana et al., 2021).

To optimize the formulation of the TPS films, this paper introduces an innovative approach that employs different machine-learning techniques like Bayesian optimization (BO) with an adaptive design of experiments (DoE). Gaussian process regression (GPR) is used to model the data and guide the BO process (Valladares, 2020). This method is groundbreaking since the optimal formula of TPS films is estimated to be reached more efficiently and faster due to applications of machine learning software. This software predicts the next testing points based on a trendline that the computer observes (Frazier, 2018). Varying the plasticizer concentration in a water and potato starch mixture allows the film's mechanical properties, primarily elongation at break and tensile

strength, to be visualized and modified. As this research has seen so far, tensile strength and elongation at break are inversely related, so an optimal point where they are both maximized is of great interest.

The preliminary results for this study suggest optimal plasticizer concentrations, and the research presents the potential for eco-friendly TPS films with improved properties, offering a sustainable alternative to petroleum-based plastics in many different applications. Further research in the TPS field using an adaptive design of experiments and machine learning will explore different factors, such as varying the concentration of plasticizer and potato starch, and additives like calcium chloride to refine the TPS film's mechanical properties. Additional research will also analyze the effects of multiple factors at a time on the TPS film, seeing if there is a correlation between strength and elasticity that can be maximized within the design space.

PROPOSED USE OF THE POTATO STARCH

The plastic film that we are aiming to improve utilizes potato starch to be converted into TPS film for applications in the food packaging industry. This TPS film is more improved than the traditional petroleum-based plastic currently on the market, in that it is renewable and eco-friendly. Our plastic film is compostable and created with all food-safe ingredients, meaning that it will not decompose and leave toxic chemicals behind. Additionally, our TPS film has a degradation rate like that of cellulose (Zhang et al., 2014). This means that the TPS is biocompatible, making it suitable for various applications involving contact with food or sensitive materials, like plastic film for food (Ncube et al., 2020). TPS films have a lower carbon footprint due to their production methods requiring significantly less energy and methane than traditional plastics (Pavon et al., 2021). Lastly, these films can be utilized for many different applications based on their specific mechanical properties, primarily their high tensile strength and elongation at break. They can achieve varying levels of flexibility, moisture resistance, and strength, making them suitable for applications in liquid deposition modeling (LDM) and other additive manufacturing processes.

COMPOSITION OF THE TPS FILM

The TPS film is made of three main ingredients: potato starch, plasticizer, and water. The potato starch is the primary component and allows the long chain of glucose molecules within the starch to cross-link, forming the biopolymer film structure (Collier et al., 2022). The plasticizer we used is composed of glycerol and white vinegar (5% acetic acid). Water, the final ingredient, acts as a solvent to allow the starch and plasticizer to dissolve uniformly for a more homogeneous solution. Water also facilitates the gelatinization process of the potato starch during production and the breakdown of the starch, creating a mixture that can be poured to take the shape of the mold that it is in.

For our film, we aimed to vary the plasticizer concentration to see if there would be an optimal point where both elongation at the break and maximum strength are maximized. This was a one-factor design of experiments, meaning that the wt% of one factor was varied (plasticizer) while other factors, such as the amount of water and the boiling time, remained constant.

TAILORING OF THE TPS FILM PROPERTIES

To tailor the tensile strength and elongation at the break of the TPS film, our research group developed machine learning methods, primarily GPR and BO, to discover the optimal weight fraction of the plasticizer for the TPS composition. These machine learning methods use different kernels for the GPR to minimize the effects of the noisy data, but for the results in this paper, the Matern 32 kernel was utilized.

GPR is a machine learning technique to solve regression problems by defining a probability distribution over infinitely many possible solutions (Valladares et al., 2020). Therefore, instead of modeling a specific function, it models the distribution of functions that could describe the data. As the output, GPR provides a probabilistic estimation of the model describing the data, which allows us to compute empirical confidence intervals and make decisions regarding the optimal TPS composition within a BO framework.

BO is an iterative search process grounded in Bayesian inference. It aims to find designs or inputs likely to optimize an objective function (Packwood, 2017). In this research, the objective function corresponds to the desired properties of the TPS film, such as the ultimate tensile strength and elongation at break. BO utilizes the probabilistic outputs of GPR, including the mean and variance functions, to construct an acquisition function (AF). This AF balances exploiting known promising regions (based on the mean) and exploring uncertain areas (based on the variance). By iteratively evaluating the acquisition function, BO guides the search toward the optimal composition of the TPS film, enabling efficient and effective optimization. Our work implements various AFs, including the statistical upper bound of the GPR, expected improvement, and knowledge gradient. Combined with an adaptive DoE, the use of GPR within a BO framework successfully modifies the exploration of the TPS composition to find an optimal design efficiently.

METHODOLOGY

This research employed an adaptive DoE as opposed to a traditional DoE to have testing points that are centered around the trends that the data is exhibiting (Myung et al., 2013). Our testing procedures involved testing the same control point for every experiment, and then testing additional points that both the GPR mean function and AFs predicted would present the most improvement. Five formulas of TPS films were tested for the first iteration, and 9 formulas of TPS film were tested for the second. Each of these 14 formulas was tested 5 times for a total of 70 films. We then averaged the 5 values collected from testing each formula and used this value to compute our mean and uncertainty functions. By following this process, our testing points were tailored to the material properties. This makes researchers believe that this will help us reduce the total number of experiments needed to reach a maximum point.

GAUSSIAN PROCESS REGRESSION WITH NOISY DATA

GPR is a mathematical machine-learning technique to solve regression problems by defining a probability

distribution over infinitely many possible solutions (Zhikun et al., 2013). Therefore, instead of modeling a specific function, it models the distribution of functions that could describe the data. This makes Gaussian processes (GPs) flexible and better suited to handle complex and nonlinear relationships between data points. In short, GPs are basic supervised learning methods that aim to solve regression and probabilistic classification problems.

A GPR model is defined by two key components: a mean function and a covariance function (also known as a kernel function). The mean function represents the expected value of the function at each point, while the covariance function determines how the function values at different points in the input space are related to each other.

The covariance function plays a crucial role in a GPR. It captures the similarity or correlation between different input points and assigns higher values to points expected to have similar function values. By leveraging this covariance structure, the Gaussian process can make predictions and generate new function values based on the observed data at unobserved points.

One of the advantages of GPRs is that since the prediction is probabilistic, one can compute empirical confidence intervals and make decisions based on those about whether one should refit the prediction model in a specific region. Additionally, GPs work well for small to large data sets, even though GPs can be computationally intensive.

Some disadvantages of these GPs are that they use the whole sample or feature information to perform the prediction and tend to lose efficiency when the space is multidimensional. In our approach, the data is corrupted by noise; rather than observing exact measurements y , it observes

$$z = y + \varepsilon \quad (1)$$

where ε is a vector of random errors independent of y . If we are not able to observe y exactly, we then have some added Gaussian noise within our data. Observations of an objective function are typically corrupted by this noise due to limitations of measurement or statistical

approximation. In many real-world scenarios, noise will be present, so it is important to be able to handle these noisy observations to maximize the utility. After conditioning GPs on data corrupted with noise, as the noise increases, we can create different levels of credibility, so the observations have less influence on our beliefs. As these measurements in Eq. (1) are assumed to be variations from the true function, the posterior mean is not compelled to interpolate perfectly through the observations as the model would do with no noise present.

BAYESIAN OPTIMIZATION

BO is a helpful tool in black-box optimization, especially for problems with a limited number of function evaluations. It is an optimization method that updates a surrogate model and uses an acquisition function to determine the solution that would give the best additional information value when added to the current data set.

BO typically uses a GPR to predict the function that needs to be optimized. One reason for this is that these GPs can estimate the uncertainty of the prediction at a given point. Once the uncertainty is evaluated, the model is then able to estimate the possible gains at the points that are unknown. After each query, the AF is reevaluated, and a new query is chosen to maximize the AF. The most used acquisition function is called expected improvement (EI), defined by

$$\begin{aligned} EI(\mathbf{x}) = & (\mu(\mathbf{x}) - f(\mathbf{x}^+))\psi\left(\frac{\mu(\mathbf{x}) - f(\mathbf{x}^+)}{\sigma(\mathbf{x})}\right) \\ & + \sigma(\mathbf{x})\phi\left(\frac{\mu(\mathbf{x}) - f(\mathbf{x}^+)}{\sigma(\mathbf{x})}\right) \end{aligned} \quad (2)$$

where $\mu(\mathbf{x})$ and $\sigma(\mathbf{x})$ are the mean and variance of the GP regressor at \mathbf{x} (respectively), f is the function to be optimized with an estimated maximum at \mathbf{x}^+ , and $\psi(z)$, $\phi(z)$ denotes the cumulative distribution function and density function of a standard Gaussian distribution (Garnett, 2023). EI measures the expected improvement over the current best solution in terms of the objective function value. Additionally, it evaluates the potential of a candidate solution by comparing its performance against the current best solution and the uncertainty associated with the objective function. It focuses more on exploration, or exploring areas of the search space where there is

a higher probability of finding better solutions than the current best.

The knowledge gradient (KG) is another AF that can quantify the expected increase in the maximum of the modeled black-box function f from obtaining additional random observations collected at the candidate set \mathbf{x} . Adopting the definition from Garnett's book *Bayesian Optimization*, the knowledge gradient AF is defined as

$$\alpha_{KG}(\mathbf{x}; \mathcal{D}) = \int [\max_{\mathbf{x}' \in \mathcal{X}} \mu_{\mathcal{D}'}(\mathbf{x}')] p(y | \mathbf{x}, \mathcal{D}) dy - \max_{\mathbf{x}' \in \mathcal{X}} \mu_{\mathcal{D}'}(\mathbf{x}') \quad (3)$$

where $\mu_{\mathcal{D}'}$ is the posterior mean of the prediction in the dataset \mathcal{D}' for the entire domain \mathcal{X} . The KG AF aims to maximize the expected improvement of the objective function and quantifies the potential of gathering additional information at different points in the search space.

Typically, the KG AF shows improved BO performance with simpler acquisition functions like the previously mentioned EI AF. However, the exact computation of the KG AF is more costly than EI, due to the maximization within the expected value. While KG quantifies the value of information gathered from evaluating the candidate solutions, EI assesses the hypothesized gain over the current best solution. These two different acquisition

functions have different approaches to balancing the exploration and exploitation of the data space in the optimization process.

ADAPTIVE EXPERIMENTAL DESIGN

The adaptive DoE adopted in this study uses a GPR model within a BO framework (Figure 1). An initial dataset is obtained by varying a single factor, in this research, plasticizer content. Observations result from tensile testing of TPS films using a UTM machine. The resulting data collected, tensile strength and elongation at break, is utilized to train a GPR model. Our model utilized the Matern 32 kernel using GPflow (The GPflow Contributors, 2023). The statistical output of the model defines AFs such as EI and KG, as well as a statistical upper bound (SUB) and GPR mean. New TPS film formulations are obtained by maximizing the AFs and adding them to the sampling plan. This process continues until no further formulations can be obtained from the AFs and the prediction for improvement at each point is low.

Each TPS film was made with varying amounts of plasticizer (white vinegar with 5% acetic acid and glycerin), in a potato starch and water mixture. Throughout the

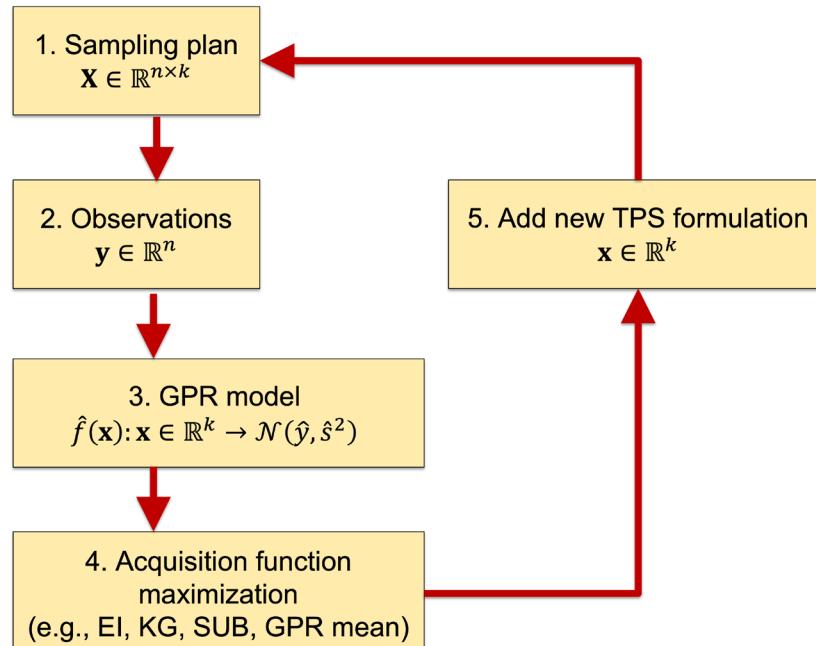


FIGURE 1. Adaptive experimental design using a Bayesian optimization framework.

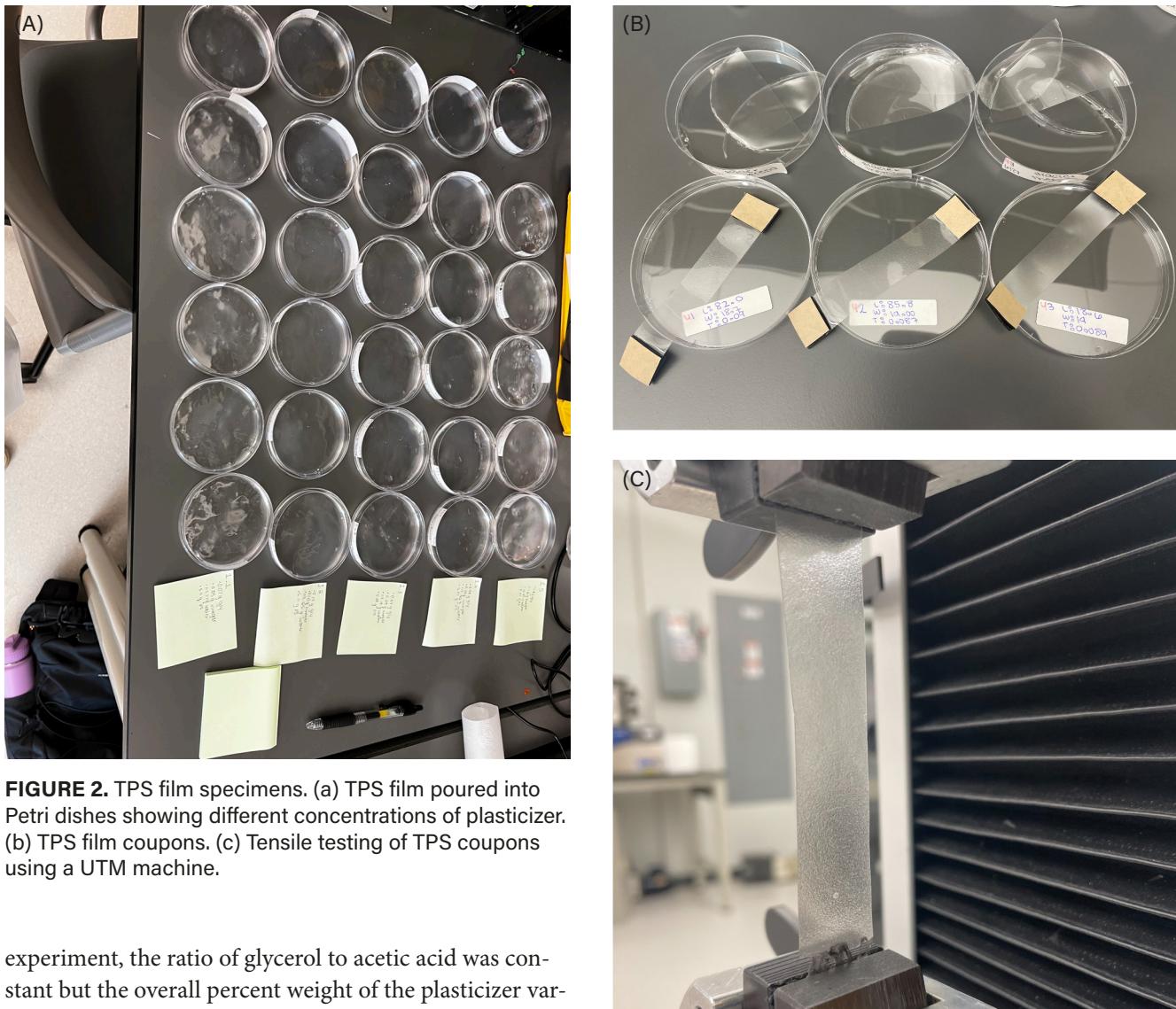


FIGURE 2. TPS film specimens. (a) TPS film poured into Petri dishes showing different concentrations of plasticizer. (b) TPS film coupons. (c) Tensile testing of TPS coupons using a UTM machine.

experiment, the ratio of glycerol to acetic acid was constant but the overall percent weight of the plasticizer varied from 0.30% to 5.00%. Each sample was boiled until 80 °C and then centrifuged for 2 minutes. Twelve grams of the TPS mixture were weighed, poured into a petri dish (Figure 2a), and allowed to dry for 48 hours. Once dry, samples were cut according to ASTM standards D882 (Figure 2b) and tested in a universal testing machine (Figure 2c) (ASTM International, 2010). Since our data is noisy, each batch had five testing points within it where the mean and standard deviation were recorded. The elongation and force applied to the sample were recorded.

RESULTS

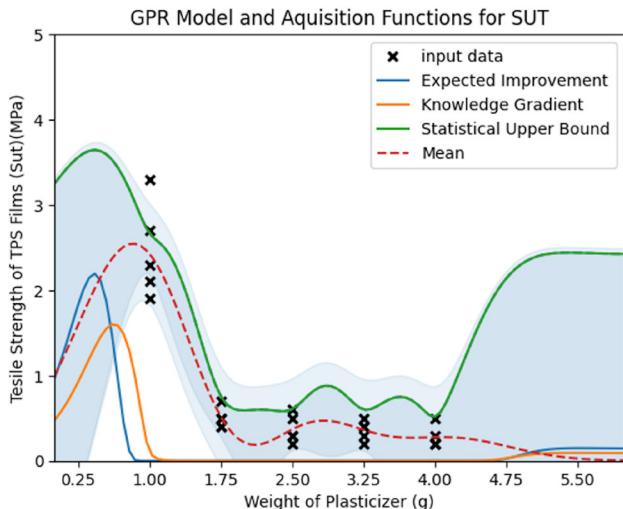
The mean value of GPR models predicted the greatest tensile strength (2.53 MPa) at 0.81 wt% plasticizer and

the greatest elongation at break (37.42%) at 3.13 wt% of plasticizer (Figure 3a and b). These values do not consider the uncertainty of the models. Other AFs, such as EI, KG, and SUB consider the uncertainty and mean values. The maximization of these AFs leads to eight new TPS designs, which are added to the dataset. The resulting GPR models after the addition of the new designs are shown in Figure 4. These designs, along with the control point are tested. The results are summarized in Table 1.

DISCUSSION

The findings of this study were that the maximum tensile strength was $4.01 \text{ MPa} \pm 1.44$ at 0.30 wt% of plasticizer

(A)



(B)

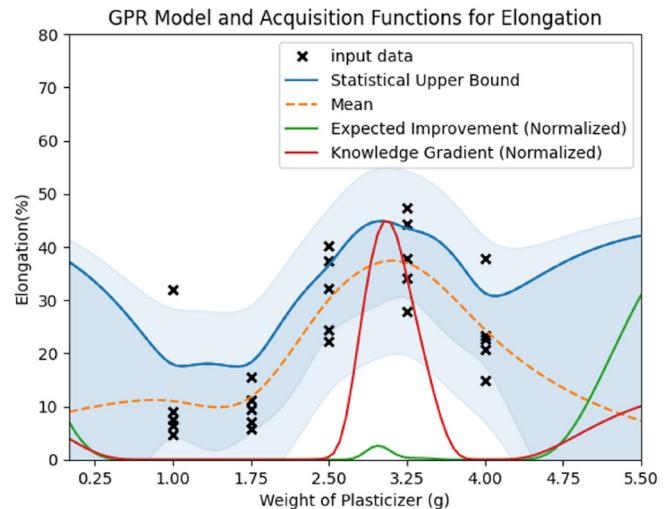
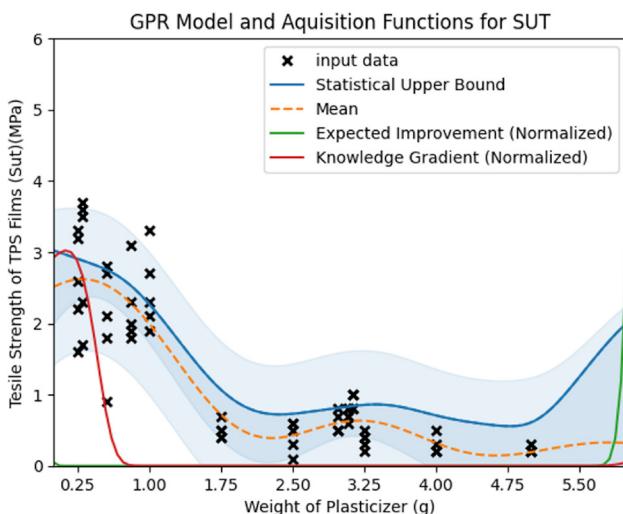


FIGURE 3. Gaussian process regression model for (a) tensile strength of the initial dataset, (b) elongation at break of the initial dataset.

(A)



(B)

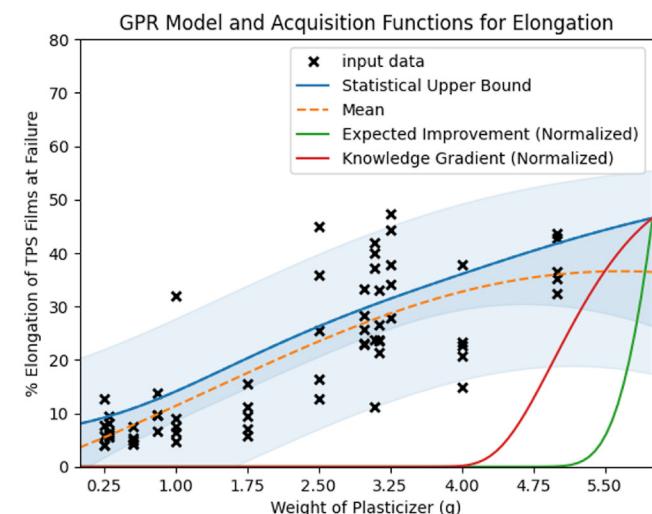


FIGURE 4. Gaussian process regression model for (a) tensile strength of the augmented dataset, and (b) elongation at break of the augmented dataset. The augmented dataset includes nine additional training points based on the acquisition function maximization.

and the maximum elongation at break was recorded at $38.12\% \pm 4.84$ at 5.00 wt% of plasticizer. One other paper in the field reported that TPS film typically has tensile strength below 5 MPa and an elongation at break of less than 50% (Zhang et al., 2003). Our findings were consistent with these results, but we believe that future iterations of the Gaussian process regression model and using additives like calcium chloride can help us exceed these previous markers significantly.

Our research effort aims to develop a TPS film formulation with mechanical properties comparable to the traditional petroleum-based plastic bags, which are predominantly used in today's market. These conventional bags have tensile strength ranging from 17.42 to 25.95 MPa and a typical elongation at break in the range of 121% to 413% (Kim et al., 2008; Radini et al., 2017). To replace these bags, the mechanical properties of the films not only need to achieve the same benchmarks of strength

TABLE 1. Varying formulas for each batch of TPS films, including the initial dataset (Batch 1.1–1.5) and the new designs based on the acquisition function testing points (Batch 2.1–2.9). The data for tensile strength and elongation at break include mean and standard deviation values (uncertainty bounds). The highest observed values are shaded.

Batch	Plasticizer (wt%)	Acquisition Function	Tensile Strength (MPa)	Elongation at Break (%)
1.1	1.00	—	2.48 ± 10.55	11.94 ± 11.34
1.2	1.80	—	0.50 ± 0.12	9.79 ± 3.88
1.3	2.50	—	0.38 ± 0.15	31.28 ± 7.82
1.4	3.30	—	0.36 ± 0.09	38.33 ± 7.80
1.5	4.00	—	0.28 ± 0.11	23.88 ± 8.47
2.1	0.30	EI SUT	4.01 ± 1.44	7.13 ± 1.68
2.2	0.25	SUB SUT	2.57 ± 0.72	6.86 ± 3.62
2.3	0.56	KG SUT	2.09 ± 0.86	5.27 ± 1.33
2.4	0.81	MEAN SUT	2.46 ± 0.98	9.89 ± 2.56
2.5	2.50	CONTROL	0.42 ± 0.21	27.10 ± 4.31
2.6	2.98	SUB ELO	0.67 ± 0.13	27.69 ± 4.25
2.7	3.08	KG ELO	0.7 ± 0.05	30.78 ± 13.00
2.8	3.13	MEAN ELO	0.91 ± 0.12	25.65 ± 4.54
2.9	5.00	EI ELO	0.22 ± 0.05	38.12 ± 4.84

and elongation but also add something that these widely used bags lack, like the ability to be composted. While we acknowledge that we are still far from surpassing these mechanical goals, the application of an adaptive DoE and the utilization of the previously mentioned machine learning methods bring us closer to this goal than ever before.

This research had some limitations in that we only analyzed one factor at a time with our adaptive design of experiments and did not specifically look at the intersection of varying percent weights of plasticizers and other factors. This is an area we hope to expand on in the future, and we plan to test multiple factors to further the experimental design space. Another limitation of this work is that with the adaptive design of the experiment, we only completed two iterations of the Bayesian optimization cycle. Ideally, we would repeat this cycle until the model converges and suggests less than 1% improvement predicted.

CONCLUSION

The research aimed to develop an eco-friendly TPS film with enhanced tensile strength and elongation at break

compared to traditional petroleum-based plastic film. Using an adaptive DoE, it was found that lower plasticizer content led to higher tensile strength, with values of plasticizer concentration below 0.3 wt%. Additionally, it was observed that higher plasticizer content resulted in greater elongation at break, with values of plasticizer concentration above 5.0 wt%. The data displayed that the EI AF most accurately predicted the optimal plasticizer concentrations, but future work is needed to test additional BO iterations until the TPS film design converges. In the future, our group aims to explore additional factors like varying the percent weights of both plasticizer and potato starch to provide more insight into the interactions between the two factors, using additives such as natural fibers and calcium chloride, and increasing mechanical properties.

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