Investigating experimental process conditions in automated robotic food manufacturing

DOE Report - Fall 22 - Group A

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Abstract
This study aims to automate the process of dairy beverage formulation and integrate design of experiment techniques. A robotic setup with integrated pH sensors was developed to perform continuous quality assessment of the process. The impact of process parameters on measured pH, used as a proxi for taste, was quantified to optimise future experiments and to explore the impact of variance in process parameters. The hypothesis of a linear model was first tested using ANOVA, and temperature was found to have a significant impact on the pH of the milk. Based on prior screening experiments, the type of powder and temperature were postulated to influence all other parameters whilst mixing time and powder concentration would be unrelated. Half effects and relative half effects of all factors were computed, and significant coefficients were chosen for further analysis. The effect of the temperature and powder concentration was found to be significant, whilst that of mixing time was challenging to accurately capture. Finally, a quadratic model with a p-value smaller than 1% was used to estimate the optimum process conditions. Overall, the study demonstrated the potential of automating dairy beverage formulation using robotic setups and DOE techniques to achieve precise and consistent results. Future work can explore the application of the method to other food products and the integration of more complex DOE designs.

Keywords: *design of experiments, robotic automation, food science*

1 | Introduction

Food science is a discipline with significant potential for automation, particularly in the area of food engineering and product optimisation [1], [2]. Currently, much of the recipe or product optimisation process is performed manually [3], [4], which is a time-consuming and labor-intensive process that limits the number and throughput of trials, as well as providing only sparse data regarding the process. Prior works on the beverage domain have focused on automation of individual sensors for food optimisation [5] and exploration of food flavor [6], but there has been limited demonstration of this approach applied to larger-scale and longer-lasting processes performed by the robotic system. By utilising robotic automation in the formulation of dairy beverages, we can optimise the experimental process for both time and precision, and compare the performance of the robotic setup to that of a human.

The pH value of dairy beverages is an important quality parameter as it is closely related to the taste and consistency of the product [7]. Any variation in ingredient quality or quantity, including modification of preparation parameters, can lead to variation in the measured pH, making pH adjustment essential in maintaining product consistency. In order to maintain product quality, adjusting the taste to the nominal range is important in both food formulation and mass manufacturing processes. By developing a robotic setup with integrated pH sensors, we can perform continuous quality assessment of the process.

Design of experiments (DOE) can greatly benefit the field of food science by allowing for a systematic exploration and optimisation

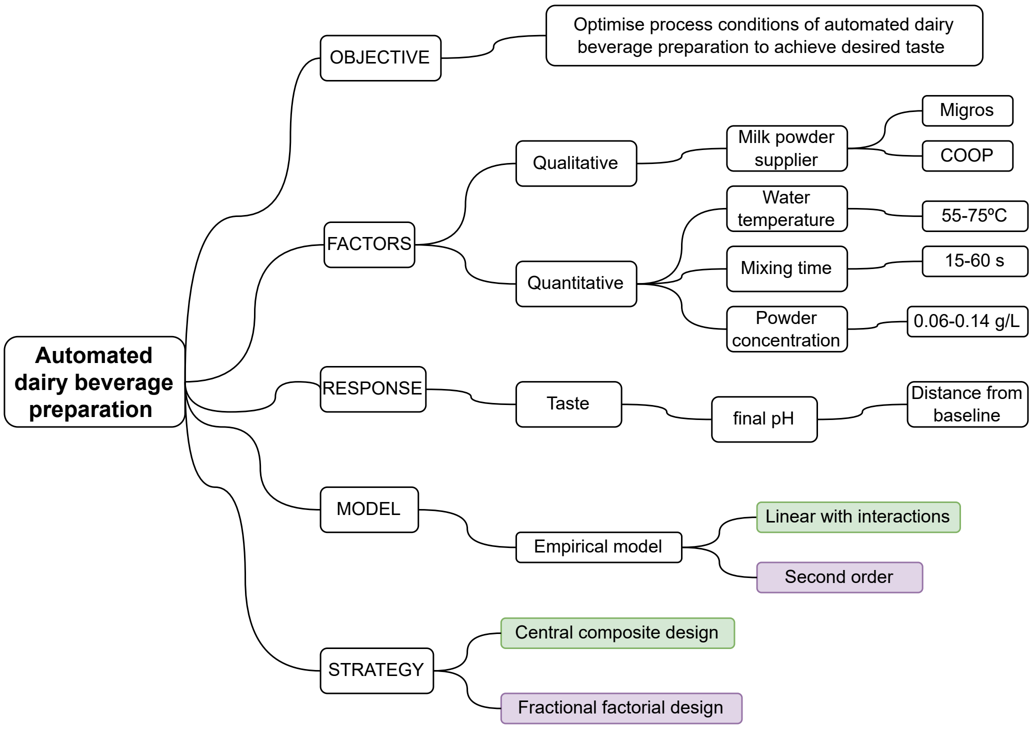

Figure 1.1: Project mindmap

of the experimental process. Barroso et al. uses a central composite design (CCD) to optimise the brewing parameters for coffee to enhance its aroma and taste [8]. Sonawane et al. uses response surface methodology (RSM) to optimise the formulation of probiotic apple juice [9]. Despite the potential benefits of DOE in food science, it has not been previously used in combination with robotic automation. By utilising a robotic setup for dairy beverage formulation and integrating DOE techniques, we can quantify the impact of process parameters on measured pH, optimize future experiments from a time, precision, and accuracy perspective, and explore the impact of variance in process parameters in terms of precision, accuracy, and reliability of sensory measurements.

We find in this analysis the factor that do and don't have significant impacts on the pH. We find also that the quadratic model captures the effects of the variables better than the linear model with interactions. In this report, we first present the project mindmap (Figure 1.1) that summarises our choice of DOE techniques and analysis. Then, the experimental setup, plan and factors and response are presented. We finally discuss our results, after which we will conclude with suggestions for future experiments.

2 | Methods

2.1 Selection of factors and response

We have identified four main factors affecting the dairy beverage preparation, summarised in Table 2.1. The type of milk powder is the only discrete variable in our analysis. We performed experiments with two different powders from two suppliers (Migros and Coop). The temperature of the water is controlled with a heating mat and measured with a temperature probe within a range recommended by industry and from literature [10]. The cooling rate will depend on the outside temperature and the time of the whole process (from the dispenser to the final temperature probe). Mixing is accomplished by a kitchen mixer [11] and programmatically controlled. No factors have been recognised to be difficult to vary. Previous experiments have shown the close link between mixing speed and powder concentration. From those experiments, we concluded that we should only consider one mixing speed for the selected concentration range. The uncertainty of the temperature probe is 0.5ºC, while the measurement error of the scale is 2g.

Table 2.1: Factors

Item	Nature	Range	Unit	Measurement
Milk powder type	Qualitative	Two suppliers	NA	NA
Water temperature	Quantitative	65-75	°C	Temperature probe
Powder concentration	Quantitative	25-75	g	100 kg load scale
Mixing time	Quantitative	15-60	s	Python program

Our objective is to improve the taste of the dairy beverage, which is not easily measurable and subjective to external factors. The final pH of the drink will be used as a proxy measure of taste [7]. Our industrial partner has conducted large scale tasting panels and concluded that the final pH of the dairy beverage should be between 6.5 to 6.7. Moreover, for each powder type, we performed a fully manual preparation following the instructions on the milk powder packaging. The pH for this mixture, which was also tasted and approved by human volunteers, will be regarded as the baseline. Indeed, our aim is to optimise the automated set-up to achieve a milk powder as tasty as if it was manually prepared by the consumer. pH will be experimentally measured with a probe with a tolerance of +/- 0.05 [12].

2.2 Experiment matrix

Screening of the various process variables has previously been done and we will focus here on understanding the relative significance and impact of those selected factors on taste to optimise the process. An essay matrix was designed to maximise the amount of information collected with the minimal number of experiments. First we tested the two levels (min and max), coded as -1 and 1, for the three continuous variables. We then added 10 points at intermediate levels: 7 points changing only one parameters and 3 points varying two variables. Finally, we add a centre point which is replicated for both powders. Our design resembles a fractional 2^k factorial design with manually added points of interest. Once the list of experimental conditions was set, we randomly assigned a powder type to each point. The random assignment was repeated until the distribution was deemed suitable (Figure 2.1). By doing so, we avoided the repetition of the full matrix of experimental conditions for both powder types. This design will enable us to infer the constant parameters of both linear models with interactions and quadratic models. Before performing the experiments, our design was validated by the commercially available JMP software for DOE.

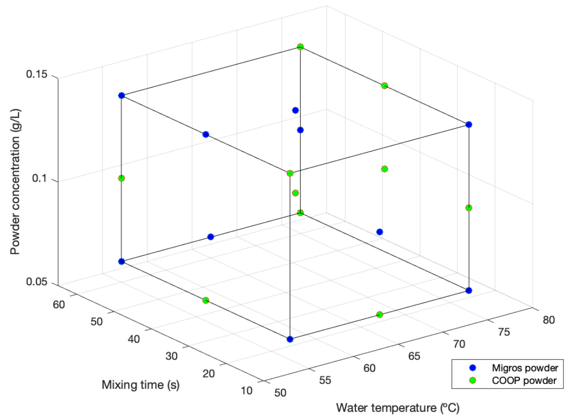

Figure 2.1: Graphical representation of the experiment matrix

2.3 Experimental set-up

The customised robotic setup is shown in Figure 2.2 and the experimental process consists of four main stages: water filling, pouring of dairy powder, mixing of the ingredients with an over-head mixer, and finally measurement with a pH probe. Following manual weighing of the powder for improved accuracy, the sample is automatically transferred from one process step to the other with a UR5 robotic arm. This experimental set-up enables a high through-put generation of results (circa 10min per data point).

3 | Results and Discussion

3.1 ANOVA with qualitative factors

Our first analysis considers the hypothesis of a linear model without any interactions and with constant coefficients. We use the Matlab function *anovan()* to perform the ANOVA and infer the coefficients. From Figure 3.1, it is clear that the temperature has a significant impact on the pH of the milk. Temperature also displays the lowest p-value, further attesting of its significance. Smaller concentrations than the recommended value result in a greater distance from the baseline pH and thus a bad taste. With the higher p-value (60.95%), mixing time plays a minor role in the final pH of the

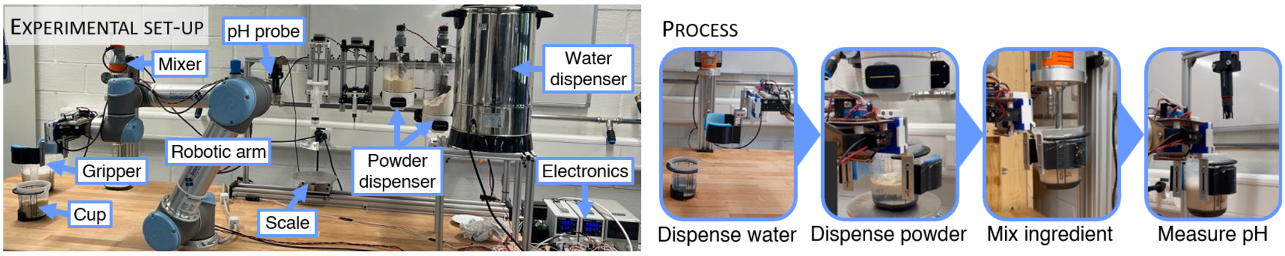

Figure 2.2: Left: Customised automated robotic setup for food science processes. **Right:** Experimental process for dairy beverage making and pH measurement.

dairy beverage. The effects for 15 and 37.5s mixing are very similar, confirming that it is not a critical parameter in the preparation of the milk. Finally, both powders are equivalent in terms of distances from their baseline pH.

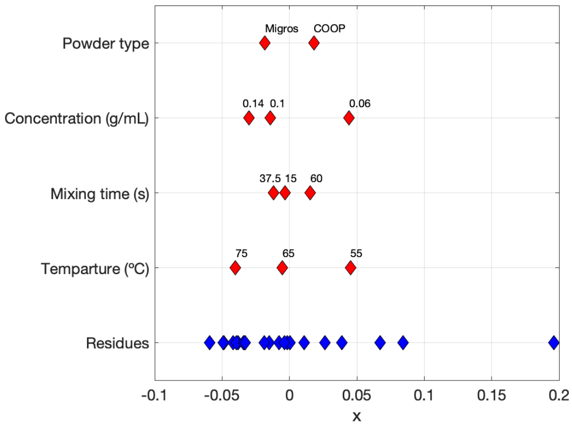

Figure 3.1: ANOVA dotplot

3.2 Determination of important interactions

Based on prior screening experiments, we postulated that the type of powder and temperature will influence all other parameters whilst mixing time and powder concentration would be unrelated. To verify those assumptions, we compute the half effects and relative half effects of all factors. Here and onward, the following subscripts are used: T for temperature, t for mixing time, C for powder concentration, and P for powder type.

The constant, a_0 is computed to be 0.123, and the barplot showing the relative half effects for the main and interaction effects are shown as Figure 3.2. The normal plot shown as Figure 3.3 also helps disqualify certain coefficients that align closest to the red line.

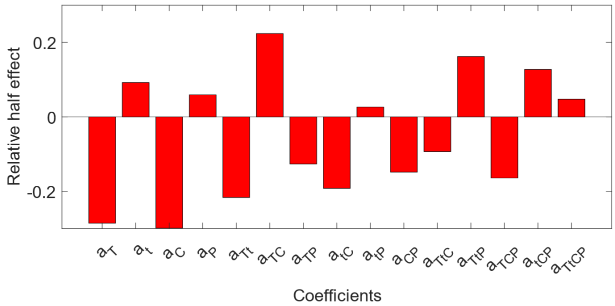

Figure 3.2: Relative half effects of all factors

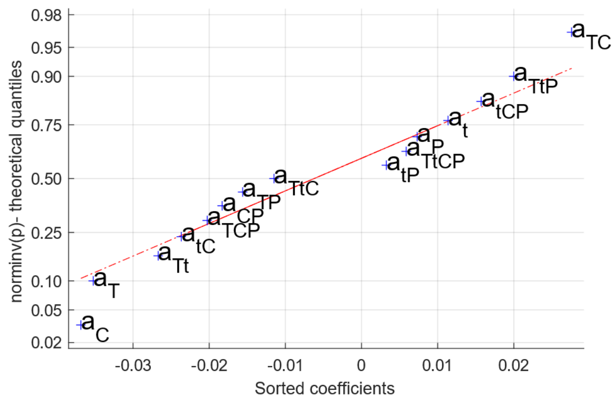

Figure 3.3: Normal plot of effects

As significant coefficients, we chose a_{T}, a_{C}, a_{Tt}, a_{TC}, and a_{tC} by setting a threshold on the relative half effect at 0.2. Additionally a_{t} was included since this term is contained in two of the significant interactions. The ANOVA was performed on the chosen coefficients and the results are summarised in Table 3.1. The effect of the temperature and powder concentration is significant because there is only 2% probability for it to be random. The effect of mixing time has a high p-value and has a large chance for it to be random, which was also shown in the barchart and the normal plot above. As for interactions both interactions including the powder concentration has a low chance for the effect to be random.

Table 3.1: Anova table for chosen significant coefficients

	SS	DF	MS	F	p
a_T	0.02	1	0.02	7.36	0.02
a_t	0.00	1	0.00	0.40	0.54
a_C	0.02	1	0.02	6.37	0.02
a_{Tt}	0.01	1	0.01	2.37	0.15
a_{TC}	0.01	1	0.01	3.28	0.09
a_{tC}	0.01	1	0.01	3.82	0.07
Error	0.05	14	0.00		

We then infer the coefficients of a linear model with the selected interactions: temperature-concentration, temperature-mixing time and mixing time-concentration. The variance inflation factors (VIF) are shown in Table 3.2. A VIF closest to 1 is optimum and similar VIF indicate that those coefficients are co-linear and that varying one or the other leads to the same variation in pH. All VIF are very close to unity (less than 10% variation), especially those for the grand mean coefficient and the coefficient related to powder concentration.

Table 3.2: Variance inflation factors (VIF) for coefficients of the linear model with selected interactions

Coefficient	VIF
Constant	1.01
Temperature	1.05
Mixing time	1.04
Powder concentration	1.01
Powder type	1.07
Temperature-mixing time	1.05
Temperature-concentration	1.02
Mixing time-concentration	1.03

One important note is that we do not fit the model to the raw pH value, instead we use the absolute distance between the measured pH and the baseline pH for each of the powders. Indeed if we were to use the raw pH value, the coefficient for the constant (a_0) would be significantly higher than the other coefficients and would mask the effects of the parameters. The pH is a log scale of the concentration of protons in solution and therefore the range of variation of the pH is relatively small, although the [H^+] has changed significantly. Moreover, comparing the baseline pH with the raw pH measurement enables a fairer comparison between the two powder which have different compositions and thus different baseline pH.

3.3 Comparison Linear and Quadratic models

The coefficients for the quadratic model were inferred and are shown in Figure 3.4. Both models capture well that the effect of mixing time alone is insignificant. Increasing the temperature or the concentration reduces the distance from the baseline pH, therefore improving the taste. The quadratic term for temperature has a marginal impact, whilst that of mixing time and concentration seem important and increase the distance form the baseline pH. When fitting a quadratic model, the constant coefficient decreases, indicating that more effects are being captured by the model.

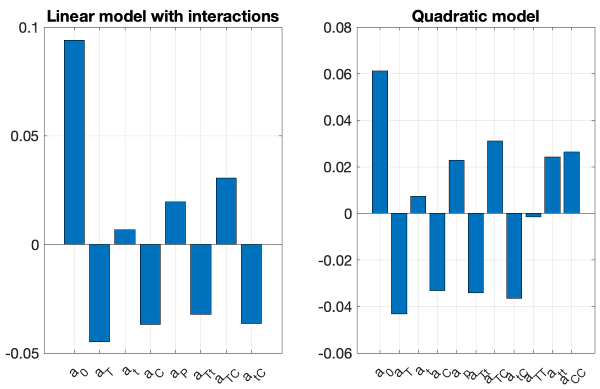

Figure 3.4: Coefficients for linear model with selected interactions and quadratic model

For both cases, the p-values are smaller than 1% indicating that the model captures the effects well since there is less than 1% chance that the observed effect of a variable is a coincidence. Moreover, the aliases of the quadratic terms are most important on the constant a_0. There are no aliases between the quadratic term for temperature and temperature itself, neither between the quadratic terms for mixing time and concentration and the coefficient for the mixing time-concentration interaction. The comparison between the linear model with interaction and the quadratic model reveals that the quadratic model provides a better fit to the data as indicated by its smaller p-value. This suggests that the quadratic model captures more of the underlying relationships between the variables and the response.

Finally, we wish to discriminate between the linear model with interaction (Z_1) and the quadratic model (Z_2). To do so, we decompose the response as follows:

$$Y = Z_1(\widehat{\alpha_1} + A \times \widehat{\alpha_2}) + (Z_2 - Z_1 \times A)\widehat{\alpha_2} \quad (3.1)$$
$$= Z_1\widehat{\alpha} + Z_{1,2}\widehat{\alpha_2} \quad (3.2)$$

The p-value for the quadratic model is 12% smaller than that of the linear model with interactions, indicating that there is less chance that an effect is fortuitous with this model.

3.4 Model optimisation

With the coefficients for the quadratic model, we can look for the optimum process conditions to minimise the distance from the baseline pH, which is assumed to be linked to a more tasty beverage. To do so, we used the Matlab function *fmincon()* to find the minimum distance from the baseine pH, constrained by the upper and lower bounds of the variables. Since our response is the absolute distance, we only consider the positive domain. The optimum conditions which enabled the smallest absolute distance from the baseline pH are displayed in Table 3.3.

Table 3.3: Optimum process conditions

Variable	Migros	COOP
Temperature (ºC)	71.3	75
Mixing time (s)	50.4	60
Powder concentration (g/L)	0.13	0.13

For the Migros powder, the optimum point results in a null distance, whilst for the COOP powder, the difference is 2%.

3.5 Suggested new set of experiments

Our analyses suggested that the effect of mixing time for our experiment had a high probability of being random. In order to attempt to find out whether any number of replicates would permit us to observe a significant difference between the highest and lowest mixing time, we performed an analysis using the concept of least significant difference from Fisher as shown in Figure 3.5. The figure shows that even at a number of replicates of 30 times, it is not enough to differentiate between the two mixing times. Furthermore, this plot was extended to include a number of replicates until 100 times, but the least significant difference never crossed the line marked at $\delta = 0.017$. This suggests that it is beneficial to get rid of the factor of mixing time completely in future studies. Other factors that may be of interest related to mixing could be mixing motion by the robotic setup or temperature of the solution while mixing.

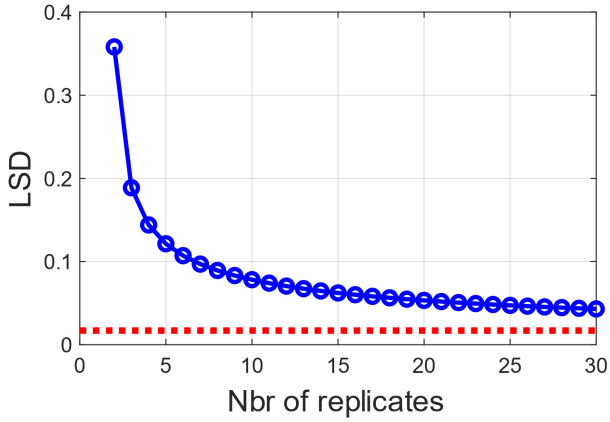

Figure 3.5: Least significant difference between the highest and lowest mixing times

For our project, we could also have resorted to the Doehlert design which is less precise but has been proven suitable for food chemistry applications [13]. For k factors, the designs are obtained from a regular k-dimensional simplex. Each variable is assigned a code with different levels, therefore enabling not only to test the bounds. Moreover, the Doehlert design is convenient to move through the experimental domain. It indeed takes advantage of the previously explored points to search for the optimal conditions of the system. However, dealing with the non-continuous variable for powder type is challenging and we would have had to perform all experiments twice: once with the Migros milk powder and then with the COOP powder. The experimental matrix in this instance for one powder is shown in Figure 3.6.

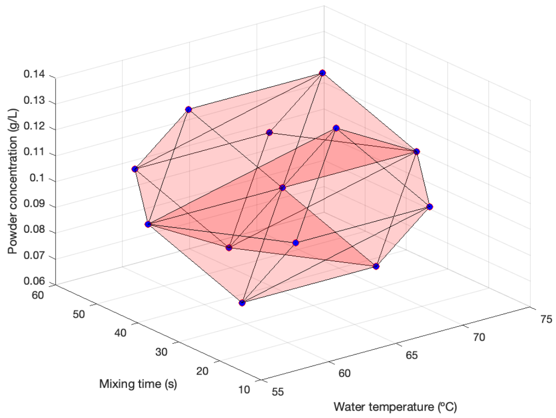

Figure 3.6: Experimental matrix for Deohlert design

4 | Conclusion

In this report, we explored the potential of utilising robotic automation in the formulation of dairy beverages and integrating Design of Experiments techniques to optimise the experimental process. By integrating robotic automation with DOE techniques, we can quantify the impact of process parameters on measured pH, optimise future experiments from a time, precision, and accuracy perspective, and explore the impact of variance in process parameters according to sensory measurements.

We identified four main factors that affect the dairy beverage preparation and chose the final pH of the drink as a proxy measure of taste. Our objective was to optimise the automated set-up to achieve a milk powder as tasty as if it was manually prepared by the consumer. We presented the experiment matrix, which was designed to maximise the amount of information collected with the minimal number of experiments.

In conclusion, our study shows that integrating robotic automation with DOE techniques can optimise the dairy beverage formulation process for both time and precision, and provide valuable insights into the impact of variance in process parameters on the final product. We also identified factors that were challenging to differentiate in the dairy beverage making process and also factors that have a high significance in the resulting pH measurement. The results of this study provide a foundation for future work in the area of food science automation and process optimisation.

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