



Migration of Cadmium Between Wrapping Paper and Food: A 2⁴ Factorial Design Approach

Muhammad, A. I., Ibrahim, I. L., Musah, M. and Mathew, J.T.

Department of Chemistry, Faculty of Applied and Natural Sciences, Ibrahim Badamasi Babangida University, Lapai, PMB 11, Niger State, Nigeria.

(*)Corresponding Author's: ibmkenci74@gmail.com, 07039142546

Abstract

The utilization of used paper as a packaging material by street food vendors is promoted as they are not expensive and easy to use. But the toxic potential of these ink stained papers has raised questions concerning the safety of these papers for wrapping food. This research seeks to investigate the migration of cadmium (Cd) from newspaper to bean cake under varying conditions, focusing on the effects of temperature (°C), contact time (minutes), food mass (g), and wrapping paper surface area (cm²) using factorial design. The samples under varying conditions were analyzed for cadmium concentration using an atomic absorption spectrophotometer. The optimum concentration of Cd was 0.075 mg/L, achieved at 65 °C, 20 minutes, 10 g, and 200 cm², with a predicted response of 0.075 mg/L and a residual value of 0.000. The results indicate that lower temperatures, longer contact times, smaller food masses, and reduced surface areas can minimize Cd migration. Statistical analysis revealed that surface area ($P < 0.05$), as well as interactions between temperature-contact time ($P = 0.027$) and temperature-mass ($P = 0.039$), significantly influenced Cd migration. While the overall model's p-value (0.066) suggests limited robustness, the R^2 value (89.15%) indicates a strong fit, with an adjusted R^2 of 67.44% highlighting potential redundancy in some predictors. These findings demonstrate that factorial design can effectively predict factors influencing heavy metal migration and provide strategies for its reduction in food packaging systems.

Key words: Bean cake, factorial design, heavy metal, migration, newspaper, variables.

Introduction

Paper is one of the oldest packaging materials, dating back to the 17th century [1]. Paper and paperboard are mostly used for packaging dry foods. Upon coating or waxing, their applications extend to the packaging and serving of wet and fatty foods [2]. These materials are frequently utilized in the production of corrugated boxes, milk

cartons, folding cartons, paper plates and cups, bags, sacks, and wrapping paper.

Today food products are wrapped and stored in newspaper, question paper and answer scripts, which contain heavy metals, these metals were attributed to the presence of additives, residues related to printing inks, adhesives or stickies, constituents arising from paper not used in food

contact applications [3]. A lot of harm is inflicted as a result of these packaging papers [4].

Migration refers to the movement of chemical compounds between the packaging film and the food it comes into contact with. This process can occur in both directions either from the packaging material to the food product or vice versa [5]. Several factors influence migration, including the type of packaging material, the nature of the food, the duration and temperature of contact, the type of interaction, the concentration of the migrating substance in the packaging, and the characteristics of the migrant itself [6]. The design is used in research that involves several factors, where it is necessary to study the interactive influence of the various factors. The design has been used by researchers such as Musah et al. [7], Gurkan et al. [8] and Al-khatibb et al. [9] to study adsorption efficiency. This research studies the migration of cadmium between newspaper and bean cake using the 2⁴ factorial design approaches. This research holds significance as it fills a crucial gap by focusing on local context (use of newspaper in packaging), providing valuable insights that were often overlooked in studies conducted in other countries.

Materials and Methods

Sample Collection and Preparation

The samples, beans which were used to prepare bean cake and wrapping paper (newspaper) were collected from Lapai market. 1 kilogram (1 kg) of beans was washed properly and grinded into paste.

Five gram of salt, 6 large pepper, 2 onions and seasoning were added to the paste. A table spoon of the beans paste was deep fried with groundnut oil. The newspaper samples were cut into 100 and 200 cm². The bean cake samples were wrapped in the newspapers, while varying the factors some physical quantities which aid migration such as temperature, contact time, surface area and sample mass as illustrated in Table 1.

To analyze heavy metal content, 0.5 g samples of bean cake were placed into digestion tubes. Each tube was treated with 15 ml of concentrated nitric acid (HNO₃) and 5 ml of concentrated sulfuric acid (H₂SO₄), which were used together to break down the organic matrix of the sample and dissolve any heavy metals present [10].

The digestion tubes were then heated using a digestion block or heating mantle, with the temperature gradually increased to 150°C and maintained for around two hours. This ensured complete digestion of the sample, breaking down organic matter and releasing heavy metals into solution. Throughout the process, the mixture was closely monitored to prevent excessive boiling or splashing [11].

Once digestion was complete, the tubes were removed from the heat source and allowed to cool to room temperature. The digested mixture was then diluted with distilled water to a final volume of 50 ml to ensure the concentration was within the measurable range for analytical instruments [12]. Filtration followed, using Whatman No. 1 filter paper to remove any solid residues, ensuring that

only the clear liquid containing dissolved heavy metals was collected. The filtered solution was then stored in clean, contamination-free sample bottles until further heavy metal analysis [13].

Sample Analysis

A PerkinElmer PinAAcle 900H Atomic Absorption Spectroscopy (AAS) was used to analyse the digested samples for the presence of cadmium.

Factorial Design for Heavy Metal Migration

The experiment employed the 2^4 factorial design for the study of the migration of heavy metal between

newspaper and bean cake. Factorial design reduces the number of experiments needed to be performed in order to achieve optimum result. Temperature ($^{\circ}\text{C}$) of food, contact time (mins) of interaction between wrapping sheet and food products, mass of food (g) and surface area (cm^2) of wrapping paper were the factors used to study the migration of heavy metal between the wrapping paper and food. The low and high levels of variables for the migration of heavy metal are shown on table 1 as follows:

Table 1: Levels of Variables for the Migration of Heavy Metal

Variables	Low	High
Temperature ($^{\circ}\text{C}$)	25	65
Contact time (minutes)	20	100
Mass (g)	2	10
Surface area (cm^2)	100	200

The required number and sequence of runs for the 2^4 factorial experiment, which examined migration factors and their two-way interactions, were determined manually. Minitab version 16 was used to predict heavy metal migration values, comparing them with actual experimental results. Meanwhile, Minitab version 21 was employed to analyze the data, determining estimated effects, main effects, P-values, F-values, and generating Pareto charts, residual plots, normal plots, and interaction plots for the responses.

Results and Discussion

Experimental and Predicted Responses for Cadmium Migration

The experimental and predicted results obtained from the 2^4 (2 by 4) factorial design for the migration of cadmium between bean cake and newspaper is influenced by temperature ($^{\circ}\text{C}$), time (mins), mass (g) and surface area (cm^2) are presented in Table 1.

Table 2: Cadmium Migration 2⁴ Factorial Experiment (response)

Run Order	Temp. (°C)	Time (min)	Mass (g)	S. A (cm ²)	Actual (mg/L)	Predicted (mg/L)	Residual
1	25	20	2	100	0.058	0.053	0.005
2	25	100	2	100	0.047	0.050	-0.003
3	25	20	10	100	0.052	0.054	-0.002
4	25	100	10	100	0.038	0.038	0.000
5	65	20	2	100	0.017	0.026	-0.009
6	65	100	2	100	0.055	0.048	0.007
7	65	20	10	100	0.055	0.049	0.006
8	65	100	10	100	0.053	0.057	-0.004
9	25	20	2	200	0.069	0.068	0.001
10	25	100	2	200	0.049	0.052	-0.003
11	25	20	10	200	0.066	0.070	-0.004
12	25	100	10	200	0.046	0.040	0.006
13	65	20	2	200	0.055	0.052	0.003
14	65	100	2	200	0.058	0.059	-0.001
15	65	20	10	200	0.075	0.075	0.000
16	65	100	10	200	0.068	0.070	-0.002

Control = 0.052±0.001

From Table 2, the optimum concentration of cadmium (Cd) in the food samples was obtained at run 15, with the highest experimental and predicted values of 0.075 mg/L each. These values were achieved under conditions of 65 °C, 20 minutes, 10 g of food, and a surface area of 200 cm². The residual value of 0 indicates an excellent fit between the experimental and predicted responses, as evidenced by the low residuals within ±0.01.

Interestingly, a higher concentration of cadmium was observed migrating from food to paper at 65 °C, 20 minutes, 2 g, and 100 cm². This suggests that reducing the mass and surface area can effectively decrease cadmium migration from paper to food.

According to Pappalardo et al. [14], the European Union has established a maximum limit for cadmium in certain food products at 0.1 mg/kg. The highest concentration recorded in this study 0.075 mg/L remains below this regulatory threshold. Nonetheless, the presence of cadmium, even at low levels, raises concerns about chronic exposure [15] and potential health risks, as cadmium is known to accumulate in the body, leading to issues such as kidney damage and bone fragility [16].

The results indicate that elevated temperatures enhance Cd diffusion from newspaper, consistent with previous findings that higher temperatures accelerate the release of contaminants from

packaging into food [17]. For instance, at 25 °C with a contact time of 100 minutes and 2 g of food, the concentration was 0.047 mg/L, which increased to 0.055 mg/L at 65 °C under similar conditions. This trend aligns with Zhou et al.'s [18] findings that increased temperatures can enhance heavy metal migration due to heightened kinetic energy that facilitates leaching from packaging materials into food [18].

The data further suggest that longer contact times generally lead to lower concentrations of cadmium in food. This observation is supported by the main effect plot indicating a negative correlation between time and concentration; as time increases, cadmium concentration tends to decrease. For example, at 25 °C with a contact time of 100 minutes and 2 g of food, the concentration was 0.047 mg/L, compared to 0.058 mg/L at 20 minutes. This phenomenon can be explained by the saturation of food with cadmium over time; initial leaching may be higher, but as equilibrium is reached, concentrations may stabilize or decrease [19]. Cakste et al. [20] also noted that prolonged exposure can lead to reduced metal concentrations due to factors like adsorption and binding with food components.

Increasing the surface area from 100 cm² to 200 cm² at 25 °C for a duration of 20 minutes resulted in an increase in cadmium concentration from 0.058 mg/L to 0.069 mg/L. This suggests that a larger surface area allows for more extensive contact between the food and wrapping material,

facilitating greater cadmium migration [21]. Research supports this finding, indicating that contaminant migration is directly proportional to the surface area of packaging material in contact with food [22].

Additionally, when comparing different masses of food at both 2 g and 10 g under conditions of 25 °C and 20 minutes, cadmium concentrations were generally higher in smaller masses (0.069 mg/L for 2 g vs. 0.066 mg/L for 10 g). This could be attributed to a dilution effect; smaller amounts may absorb a higher concentration of cadmium relative to their mass, while larger masses distribute cadmium more evenly [23]. However, at 65 °C, higher masses exhibited greater concentrations of Cd, possibly due to increased moisture content from larger food masses enhancing metal dissolution and migration [24].

Newspapers contain inks and coatings that can either promote or inhibit leaching processes depending on their chemical composition. Additives or chemicals within wrapping materials can alter interactions with heavy metals like cadmium, potentially affecting migration rates compared to unprinted or alternative wrapping materials [25]. Additionally, the fat content in foods influences heavy metal absorption due to solubility properties; since cadmium tends to bind with fats, high fat content may lead to increased absorption rates in fatty foods [26]. Given that bean cakes have significant fat content, this could further enhance cadmium migration.

Residual Plot for Cadmium Migration

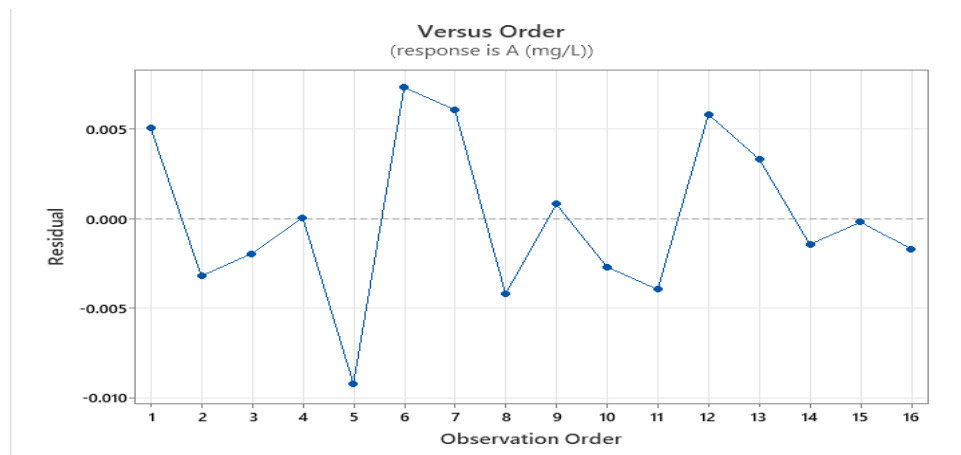


Figure 1: Number of Run Responses Plot for Migration of Cd Between Bean Cake and Newspaper

Figure 1 displays residual plots against the experimental run order, with residuals randomly scattered around zero, validating the model. Residuals, which represent the difference between experimental and predicted values [27], were within ± 0.01 for cadmium (Cd) migration, indicating a

strong fit between the model and experimental data. The residual plots were consistent with findings from studies on Cd^{2+} adsorption by Musah et al. and heavy metal release from lead smelting slag by Gurkan et al. [8].

Main and Interaction Plots for Cd Migration

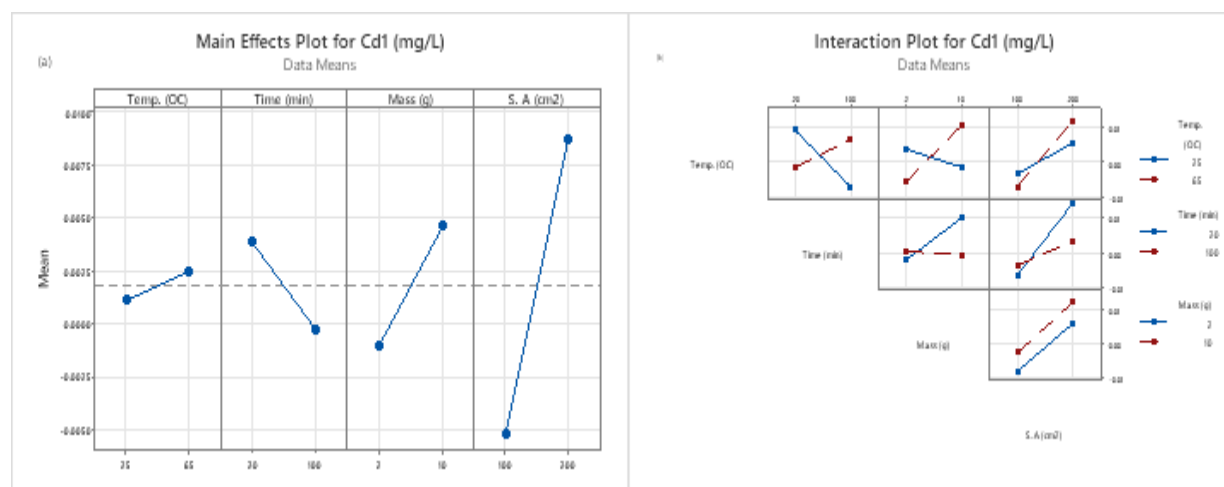


Figure 2(a & b): Main Effects and Interaction Plots

Figure 2 illustrates the main and interaction effects of factors influencing Cd migration. The factors analyzed were food temperature (A), contact time (B), food mass (C), and wrapping paper surface area (D). Figure 2a presents the main effects plot, providing a visual representation of how each factor influences migration. A near-horizontal slope indicates a minimal effect [28], and in this case temperature had a weak influence on Cd migration, while the

surface area of wrapping paper had the strongest effect on the migration of Cd between bean cake and newspaper [27]. Figure 2b depicts the interaction effects of different factors on Cd migration. If two factor lines remain parallel, there is no interaction, whereas if they cross, it indicates an interaction [29]. The interaction of temperature*contact time and temperature*mass had higher interaction effect while mass*surface area had the least interaction.

Normal Plot and Pareto Chart for Cd Migration

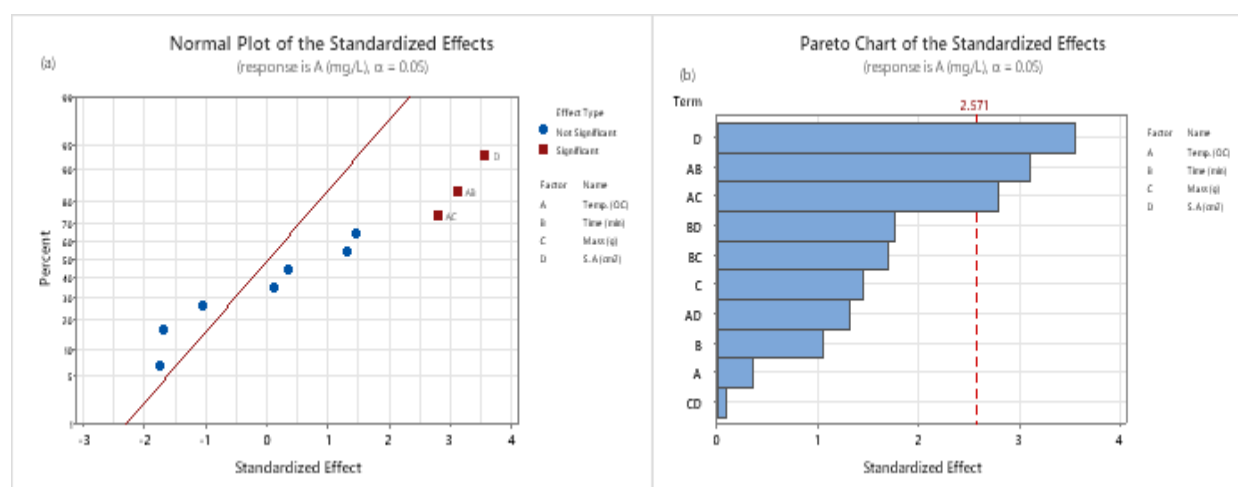


Figure 3(a & b): Normal plot and Pareto Chart for Cd Migration

The normal plot of standardized effects in Figure 3a shows that points close to the reference line represent factors with minimal influence on Cd migration, while those farther away indicate significant effects. The surface area of the wrapping paper was identified as a key factor, and interactions between temperature and contact time also had a strong influence. Another interaction with a strong influence on the

migration of Cd is temperature*mass. Factors on the positive (right) side of the line have positive influence on the migration of Cd while those on the negative (left) side of the line have negative influence on the migration process, this indicates that all the interactions (*) with influence on this plot, temperature*contact time and temperature*mass had positive influence on the migration of Cd [28,30]. Figure 3b present the

student's t-test as Pareto chart which was used to determine whether the calculated effects vividly differ from zero. At 95 % confidence level, the t-value equals 2.571 [31]. The vertical line in figure 3b represent the minimum statistically significant effect level [23] and the horizontal row in the table represents the absolute values of the Student's t-test for each effect [27]. Factors with t-values ≥ 2.571 were deemed significant for Cd migration

Estimated Effects and Coefficients for Migration Factors

Main effect, interaction effect, coefficient of the model, standard deviation of every coefficient and probability of responses for the migration of Cd are presented in Tables 3. Statistically

significant main effects and interactions are considered to be those that have their P - values less than 0.05 ($P < 0.05$). When coefficients of main and interaction effects are positive, then the uptake of the metal is favored at high value [32], high level of a factor if the effect is negative [13]. Contrarily, there will be a reduction in migration of the metal for high t-values and low P-values indicated greater significance [31]. Upon reviewing Table 3, it was observed that factors with t-values $< \pm 2.60$ and P-values > 0.05 were not significant. However, the surface area of wrapping paper and the interactions between temperature & contact time and temperature & mass had significant effects, with P-values below 0.05.

Table 3: Estimated Effects and Coefficients for Cd migration

Term	Effect	Coef	SE Coef	T-Value	P-Value
Constant		0.05381	0.00195	27.57	0.000
Temp. (°C)	0.00137	0.00069	0.00195	0.35	0.739
Time (min)	-0.00413	-0.00206	0.00195	-1.06	0.339
Mass (g)	0.00563	0.00281	0.00195	1.44	0.209
S. A (cm ²)	0.01388	0.00694	0.00195	3.55	0.016
Temp. (°C)*Time (min)	0.01213	0.00606	0.00195	3.11	0.027
Temp. (°C)*Mass (g)	0.01088	0.00544	0.00195	2.79	0.039
Temp. (°C)*S. A (cm ²)	0.00513	0.00256	0.00195	1.31	0.246
Time (min)*Mass (g)	-0.00662	-0.00331	0.00195	-1.70	0.150
Time (min)*S. A (cm ²)	-0.00688	-0.00344	0.00195	-1.76	0.139
Mass (g)*S. A (cm ²)	0.00037	0.00019	0.00195	0.10	0.927

R-Sq = 89.15 % R-Sq(adj) = 67.44 %

The findings indicate that the value of R^2 (89.15%) suggests a strong correlation between the experimental and predicted cadmium (Cd) migration, demonstrating good agreement in the response data. The analysis shows that surface

area has a positive effect on Cd migration, as indicated by a high positive t-value and a low p-value, which confirms the significance of surface area in the migration process. Specifically, an increase in surface area corresponds to an

increase in Cd migration, highlighting its critical role in contaminant transport [26].

Conversely, the negative t and effect values associated with contact time imply that longer contact times lead to a decrease in Cd migration. This suggests that as contact time increases, the availability of Cd for migration diminishes, potentially due to adsorption processes or other interactions within the food matrix [12].

Analysis of Variance for Cd Migration

Table 4 presents the analysis of variance (ANOVA) results for Cd migration, which assessed the influence of main factors and their

interactions. The results (Table 4) indicate that significant parameters and estimated migration values depend on the main effects temperature, contact time, food mass, and wrapping paper surface area as well as their two-way interactions. Among the main effects, surface area showed the highest significance ($P = 0.016$), while temperature-contact time ($P = 0.027$) and temperature-mass ($P = 0.039$) interactions also demonstrated notable effects. Similar trends were observed in Mn^{2+} adsorption studies conducted by Musah et al. [25] reported similar trends in the adsorption of Mn^{2+} using factorial design experiment.

Table 4: Analysis of Variance for Cd (mg/L) Migration

Source	DF	Adj SS	Adj MS	F-Value	P-Value
Model	10	0.002504	0.000250	4.11	0.066
Linear	4	0.000972	0.000243	3.99	0.081
Temp. ($^{\circ}C$)	1	0.000008	0.000008	0.12	0.739
Time (min)	1	0.000068	0.000068	1.12	0.339
Mass (g)	1	0.000127	0.000127	2.08	0.209
S. A (cm^2)	1	0.000770	0.000770	12.63	0.016
2-Way Interactions	6	0.001531	0.000255	4.19	0.069
Temp. ($^{\circ}C$)*Time (min)	1	0.000588	0.000588	9.65	0.027
Temp. ($^{\circ}C$)*Mass (g)	1	0.000473	0.000473	7.76	0.039
Temp. ($^{\circ}C$)*S. A (cm^2)	1	0.000105	0.000105	1.72	0.246
Time (min)*Mass (g)	1	0.000176	0.000176	2.88	0.150
Time (min)*S. A (cm^2)	1	0.000189	0.000189	3.10	0.139
Mass (g)*S. A (cm^2)	1	0.000001	0.000001	0.01	0.927
Error	5	0.000305	0.000061		
Total	15	0.002808			

F-values were used to assess the model's significance [33]. A high F-value ($F > 5.00$) and a low P-value ($P < 0.05$) indicated statistical significance of the main effects or interactions at

the 5% level. According to Table 3, the surface area had an F-value of 12.63, supporting the model's predictive accuracy. The interaction between temperature and contact time had a

positive effect, with an F-value of 9.65. This finding aligns with research suggesting that a larger surface area increases contact between food and wrapping material, thereby enhancing Cd migration [21]. Other studies have also demonstrated that contaminant migration is directly proportional to the packaging material's surface area in contact with food [20].

Conclusion

The model used in this study provides a valuable tool for assessing cadmium migration levels, especially at lower concentrations, which is critical for ensuring compliance with safety standards. Though the overall model is statistically insignificant, it shows that one of the main factors surface areas and the interactions temperature-contact time and temperature-mass are significant in cadmium migration.

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