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Method Validation and Chemometric Analysis of UV-Absorbing Contaminant Migration from Low-density Polyethylene Packaging Materials into 8% Ethanol

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***Abstract* – Ensuring food packaging safety is vital due to potential chemical migration. This study validated an extraction method using 8% ethanol, a simulant for alcoholic products per US 21 CFR Part 177, and evaluated the influence of LDPE thickness on total UV-absorbing contaminants (TACs). Twenty-two samples (6–130 µm thick) from 13 Mega Manila locations were tested. Method validation showed limits of detection (LOD) and quantification (LOQ) of 0.010 and 0.025 Au, respectively. Repeatability and intermediate precision were acceptable, with %RSDs from 8.5% to 35.8%. All absorbance values were below the Philippine FDA limit (0.300 Au), with PE-0008 and PE-0022 having the highest (0.154 and 0.123 Au). Chemometric analysis showed a moderate positive correlation between thickness and TACs (Spearman's ρ = 0.481), though Kruskal-Wallis (p = 0.2042) and LOESS regression indicated thickness alone doesn't drive migration. K-means clustering and LOESS trends suggest peak migration at 30–40 µm. Future work should apply chromatographic techniques for comprehensive LDPE safety assessment.**

Keywords: LDPE, Food packaging safety, UV-absorbing contaminants, Migration behavior, Chemometric analysis

I. INTRODUCTION

Plastics have become indispensable in modern food packaging due to their versatility, durability, and cost-effectiveness [1], [2]. Among these, low-density polyethylene (LDPE) is widely used in food contact applications [1], [3]. However, concerns over the migration of chemical contaminants from LDPE into food products have raised significant food safety issues [4] [5], [6] particularly in countries like the Philippines, where regulatory studies on migration remain limited.

This study focuses on LDPE plastic bags that are single-layer, single-use, and disposed of after use. Yet, their potential to leach harmful UV-absorbing contaminants into food has not been fully investigated. In the Philippines, LDPE plastic bags are not only used for food storage and transport but are often repurposed in alignment with Filipino culture, which values resourcefulness and thriftiness [7]. While such practices may be practical and environmentally sustainable, they also raise concerns about the potential migration of chemical contaminants into food, highlighting significant food safety issues [5], [6], [8], [9]

Although international studies on food contact materials (FCMs) have extensively evaluated chemical migration [10], these have been conducted primarily in temperate and developed countries. The Philippine context presents unique conditions—such as high temperatures, humidity [11], and variations in plastic formulations—that may influence contaminant migration differently from what has been reported in Western food safety assessments.

This study is part of a broader research initiative examining the migration of TACs from LDPE plastic bags into various food simulants. Previously, TAC migration was investigated in *n*-heptane (representing oily food products) [12] and water (representing aqueous food products) [13]. These studies established baseline migration levels under different food contact conditions. The present research expands on these findings by focusing on TAC migration into 8% ethanol, a simulant for alcoholic food products. In addition to validating an extraction method, this study applies chemometric analysis to classify LDPE materials and assess factors influencing migration behavior.

Recent studies have demonstrated the importance of evaluating the safety of plastic food packaging materials [14], especially in tropical climates like those of Southeast Asia, where higher temperatures can accelerate the

migration of harmful substances into food and beverages [15], [16]. For instance, a study in Thailand identified phthalate migration in plastic-coated food packaging materials, especially under the high-temperature conditions typical in tropical climates [15] while research in Indonesia, found plastic component migration in various packaging types exposed to different pH levels, temperatures, and organic acids [16]. These findings underscore the importance of conducting localized migration studies relevant to the Philippine setting, where LDPE bags are widely used for food storage.

The potential health risks associated with chemical migration from plastic packaging materials have been well-documented in toxicological and epidemiological research [17]. Many UV-absorbing contaminants are classified as endocrine-disrupting chemicals (EDCs) [5], which have been linked to promote hormone-sensitive disorders and cancers; produce adverse reproductive, neurological, developmental, and immune effects; as well as obesity, diabetes mellitus, and cardiovascular disease [18] [19]

The Food Safety Act of 2013 promotes a proactive farm-to-fork approach to safeguard consumer health and acknowledges packaging as a factor in preventing contamination and ensuring traceability across the food supply chain [11], [20] . While this provides a strong foundation, comprehensive policies and baseline data specifically addressing chemical migration remain limited in the Philippines [21]. Therefore, establishing a validated method and generating baseline data for UV-absorbing contaminants (TACs) in LDPE packaging is essential to support regulatory development and further strengthen public health protection. Recognizing this research gap, the project team collaborated with the Philippine Food and Drug Administration (FDA) to generate scientific data supporting future guidelines on chemical migration. This work aims to identify high-risk LDPE materials, validate a robust analytical method for TACs, and provide data-driven recommendations for regulatory decision-making and packaging industry improvements.

To address this gap, the study validated an extraction method using 8% ethanol, screened LDPE bag samples from local sources for UV-absorbing contaminants, and applied chemometric tools to analyze migration behavior. Method validation focused on limits of detection (LOD) and quantification (LOQ), and precision metrics. Absorbance data were statistically evaluated using correlation analysis, Kruskal-Wallis test, clustering, and LOESS regression to explore relationships with thickness.

The results of this study will also serve as a foundation for future research involving actual food matrices. By generating locally relevant data, this study supports national food safety strategies and risk-reduction efforts, ultimately contributing to the development of safer, more sustainable food packaging solutions in the Philippines.

II. MATERIALS AND METHODS

A. Research Design.

The study involved three main components: (1) validation of an extraction method for detecting total UV-absorbing contaminants, (2) profiling locally-manufactured and/or commercially-available polyethylene bags for UV-absorbing contaminants migrating to food simulants, and (3) application of chemometric techniques to analyze the migration behavior of LDPE materials.

B. Materials and Reagents

The study utilized monolayered LDPE bag samples obtained from Manufacturer A and local retailers. The thickness of each sample was measured using a thickness gauge. These films were tested for migration using 8% ethanol following US 21 CFR part 177 [22]. Analytical-grade ethanol and distilled water were used as solvents.

UV-Vis spectrophotometry was performed using a Shimadzu UV-1800 double-beam spectrophotometer. Absorbance measurements were recorded in the range of 220–360 nm using 5 cm quartz cuvettes in absorbance mode. The instrument was zeroed with a reagent blank prior to analysis of each sample.

Fourier Transform Infrared Spectroscopy was conducted using a Shimadzu IR-Prestige-21 equipped with an Attenuated Total Reflectance (ATR) accessory. Spectra were collected in the range of 4000–400 cm^{-1} with a resolution of 4 cm^{-1}, averaging 32 scans per sample. This analysis was used to confirm the polymer composition of the collected plastic samples as LDPE.

Other equipment included an oven incubator to maintain controlled migration testing conditions.

C. Sample Preparation and Preliminary Screening of Materials

LDPE films were cut into 5 cm × 10 cm pieces, wiped with lint-free paper, and placed in separate beakers. Extraction was conducted by fully immersing the film in 8% ethanol food simulant at a ratio of 50 cm^2 of film per 100 mL of simulant. The migration test was performed at 49°C for 24 hours. After the extraction period, the simulant containing migrated contaminants was analyzed using UV-VIS spectrophotometry within the wavelength range of 220–360 nm. From the results, samples with the lowest and highest absorbance values were identified. Additionally, a sample with an absorbance level closest to the maximum allowable limit (MAL), as set by the FDA Philippines for chemical migration into this simulant, was chosen for method validation. MAL was defined as 0.300 Au for 8% Ethanol.

D. Method Validation

The method was validated based on key analytical performance characteristics. The limit of detection (LOD) and limit of quantification (LOQ) were established to define the lowest detectable and quantifiable levels. To

determine LOD and LOQ, at least ten (10) replicates of method reagent blanks were analyzed, following the procedure for the actual test samples. While a low-absorbance sample was initially considered, erratic results at trace concentrations led to the use of method reagent blanks. This approach minimized measurement uncertainty, as reagent blanks provided a more stable baseline than low-absorbance samples, which exhibited higher variability.

The LOD/LOQ were calculated using:

$$LOD = Average\ Absorbance + (3 \times Corrected\ Standard\ Deviation) \quad (1)$$

$$LOQ = Average\ Absorbance + (10 \times Corrected\ Standard\ Deviation) \quad (2)$$

For repeatability and intermediate precision, samples with low, high, and mid absorbance levels (or those closest to MALs) were tested in at least eight (8) replicates. Two analysts were used to evaluate intermediate precision. Results were assessed using the percentage relative standard deviation (% RSD).

$$\%RSD = (Standard\ Deviation\ /\ Average\ Absorbance) \times 100 \quad (3)$$

E. Validation Criteria

Corrected absorbance values were calculated by subtracting the reagent blank absorbance from the sample absorbance. Precision and reproducibility were determined based on % RSD values.

F. Preliminary screening of LDPE materials

A total of 22 samples of LDPE were obtained from Manufacturer A and Retailers in Metro Manila. Three units of each type were tested. FTIR-ATR analysis confirmed the polymer composition before migration studies. The extractions were performed under controlled laboratory conditions, following the validated method in triplicate (n = 3). Results provided a profile of TACs across the surveyed materials. For this simulant, the absorbance values of the samples were compared against its regulatory threshold limit as implemented by FDA Philippines to assess the suitability of the materials for alcoholic food types.

G. Data Processing and Statistical Analysis

Following absorbance measurement, statistical analyses were conducted to assess trends, correlations, and variability in the migration data. Descriptive statistics such as mean, standard deviation, and range were calculated for absorbance values. Outlier detection was performed using Z-score analysis to identify extreme values that may indicate anomalous migration behavior. Kruskal-Wallis test for non-parametric comparisons of absorbance levels. A Spearman's correlation analysis was conducted to determine the relationship between thickness and absorbance. K-Means clustering analysis was used to group LDPE samples based on similarity in absorbance values. A Locally Weighted Regression (LOESS) model was applied to evaluate whether thickness significantly influenced UV-absorbing contaminant migration. All statistical computations were performed using R programming.

III. RESULTS AND DISCUSSION

A. Preliminary Screening and Identification of Representative Samples

Absorbance values across the 22 samples ranged from 0.000 to 0.154 Au (Figure 1), indicating a range of potential migration levels. Three samples highlighted in red were selected for method validation to represent low (PE-0020, 0.012 Au), mid (PE-0005, 0.093 Au), and high (PE-0008, 0.154 Au) absorbance levels.

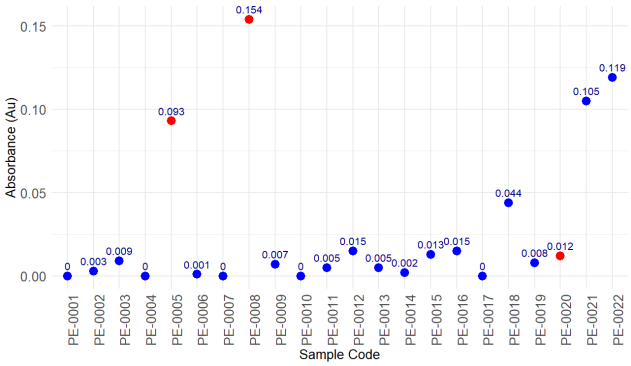

Figure 1. Scatter plot of absorbance values (TAC concentrations) across LDPE plastic bag samples.

B. Method Validation

Based on 12 replicate measurements, the absorbance values ranged from 0.001 to 0.007 Au, with an average of 0.004 Au and a standard deviation (SD) of 0.002 Au. The calculated LOD was 0.010 Au, indicating the lowest detectable absorbance, while the LOQ was determined to be 0.025 Au, representing the minimum concentration at which quantitative measurements can be reliably performed (Table 1). These values confirm that the analytical method is capable of detecting and quantifying UV-absorbing contaminants well below the MAL of 0.300 Au. The high %RSD (54.3) at low concentrations suggests that precision may improve at higher analyte levels, but given that all measured values fall below the critical MAL threshold, the method is deemed acceptable for routine analysis and regulatory compliance. The results indicate that the method successfully passed validation criteria for the detection and quantification of UV-absorbing

contaminants in food simulants.

Table 1. LOD and LOQ Results for UV-Absorbing Contaminants in 8% Ethanol

	Results
Average	0.004 Au
SD	0.002 Au
% RSD	54.3
LOD, Au	0.010 Au
LOQ, Au	0.025 Au
Remarks	< 10% MAL (0.300 Au)

Repeatability (Within-Day Precision) was assessed through replicate absorbance measurements (n=10) conducted independently by two analysts at three concentration levels using 8% ethanol. As shown in Figure 2, precision improved with increasing absorbance value. Analyst A reported %RSDs of 35.8% (low), 10.4% (mid), and 8.5% (high), while Analyst B reported 17.4% (low), 12.1% (mid), and 10.4% (high). Variability was greatest at the low level, which is expected due to signal fluctuations at trace analyte measurements. Nonetheless, mid and high levels exhibited %RSD values ≤15%, confirming acceptable repeatability.

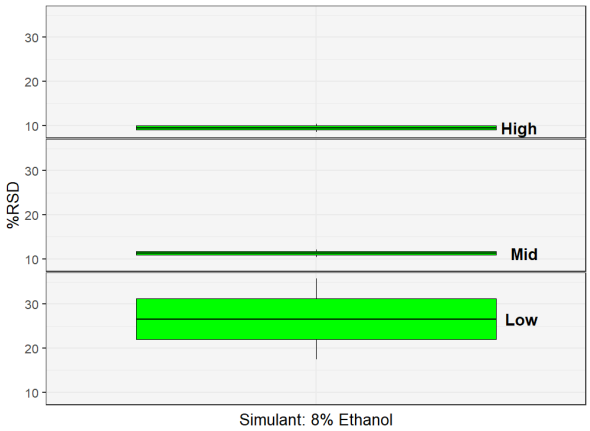

Figure 2. Boxplot comparing the % RSD for 8% Ethanol at different concentration levels (Low, Mid, and High)

Intermediate precision (Between-Day Precision), which evaluates method reproducibility across analysts and days, was computed using pooled results. As summarized in Table 3, the %RSD decreased with increasing absorbance level: 28.1% (low), 11.3% (mid), and 9.5% (high). These results indicate that the method is reproducible under varying conditions at mid and high levels, while greater variability remains at the low level, warranting further refinement for trace-level analysis.

Table 3. Results for Intermediate Precision for 8% Ethanol

Level	Absorbance (Au) Pooled Average		Pooled Average	Pooled % RSD
	Analyst A	Analyst B		
Low	0.009 ± 0.003	0.010 ± 0.002	0.010	28.1
Mid	0.079 ± 0.008	0.067 ± 0.008	0.073	11.3
High	0.111 ± 0.009	0.118 ± 0.012	0.114	9.5

The overall comparison of repeatability and intermediate precision is presented in Figure 3. The bar chart (left) highlights the observed %RSD trends, with both types of precision improving as the level increases. Notably, within-day precision (repeatability) showed greater variability at the low level compared to between-day precision, suggesting that short-term factors contributed more significantly to measurement fluctuations at trace levels. The scatter plot (right) demonstrates a strong linear correlation between mean absorbance values from repeatability and intermediate precision, underscoring the method's reproducibility. Collectively, these results validate the method's precision across different operating conditions, particularly for mid and high concentrations.

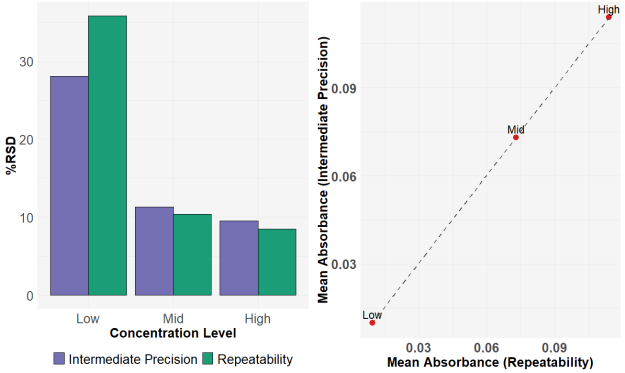

Figure 3. Comparison of % RSD for repeatability and intermediate precision (left) and correlation between mean absorbance values from repeatability and intermediate precision (right) for different concentration levels (Low, Mid, and High).

C. Establishing Acceptable Variability Criteria

Acceptance criteria were established based on the observed performance across different absorbance levels during validation. As summarized in Table 4, the %RSD thresholds reflect the expected analytical variability at each absorbance level range. These criteria provide a practical framework for evaluating data quality and determining the reliability of results during routine analysis. By aligning allowable %RSD values with absorbance-specific performance, the method accommodates natural fluctuations at trace levels while maintaining strict precision standards at mid to high levels. This approach ensures the method remains fit-for-purpose and aligned with realistic laboratory performance.

Table 4. Established Acceptable %RSD for TACs in LDPE

	Au / TACs Migrated (from 100 cm^2)	%RSD
8% Ethanol	0.010 ≤ Au	54.3
	0.010 ≤ Au < 0.073	28.1
	0.073 ≤ Au < 0.114	11.3
	0.114 ≥ Au	9.5

C. Profiling of LDPE bags and Statistical Summary

FTIR-ATR analysis was conducted to confirm polymer identity prior to migration testing. Figure 4 shows the representative FTIR spectrum of PE-0022, with strong C–H stretching bands at ~2916 cm^{-1} and ~2849 cm^{-1}, and bending and rocking vibrations at ~1471 cm^{-1} and ~719 cm^{-1}, respectively—features characteristic of polyethylene. Spectral matching returned a high similarity score (797) to LLDPE1, confirming the sample as linear low-density polyethylene. This verification ensured that only LDPE-based films were evaluated in the study.

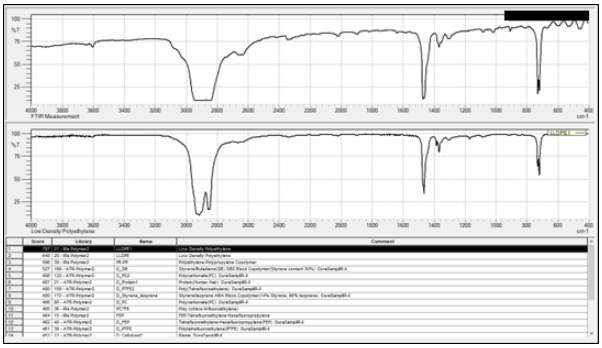

Figure 4. Representative FTIR spectrum of PE-0022

Figure 5 presents the control chart of sample absorbance. Most samples showed absorbance values below the method's LOD (0.010 Au), indicating minimal or non-detectable migration. However, several samples (e.g., PE-0005, PE-0008, PE-0021, PE-0022) showed elevated absorbance levels, though none exceeded the FDA's regulatory threshold of 0.300 Au. Descriptive statistics revealed wide thickness variability (6–130 µm), with median thickness at 22 µm (Table 5). Figure 6 shows the correlation between thickness and migration. While Kruskal-Wallis results showed no significant differences across thickness groups (p = 0.2042), Spearman's rank (ρ = 0.481, p = 0.0235) and Kendall's Tau (τ = 0.333, p = 0.0486) suggest moderate correlation. Clustering and LOESS models (Figures 7–8) indicate migration behavior may depend on material composition and processing rather than thickness alone.

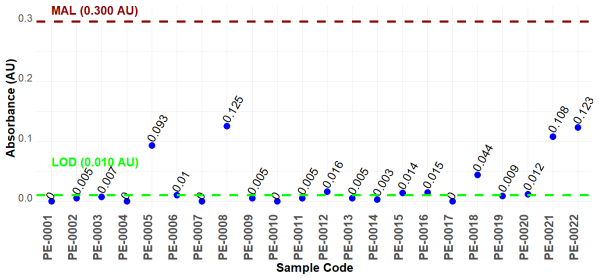

Figure 5. Control Chart for Absorbance Levels

Table 5. Descriptive statistics for film thickness (µm) and UV absorbance values of plastic samples (n = 22)

Variable	Min	1st Qu.	Median	Mean	3rd Qu.	Max
Thickness	6	6	22	29.05	24.25	130
Absorbance	0	0.0035	0.008	0.02723	0.01575	0.125

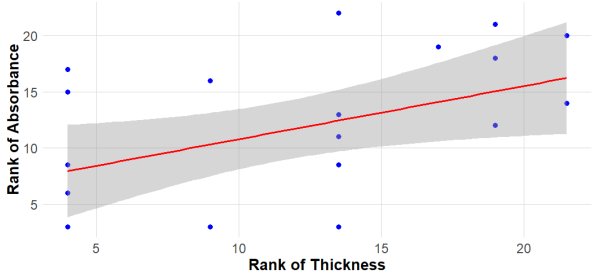

Figure 6. Spearman's Rank Correlation between Thickness and Absorbance.

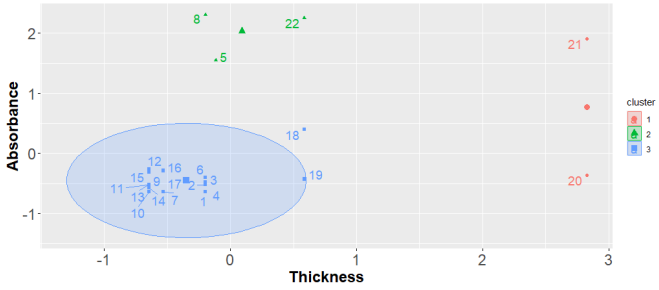

Figure 7. K-means clustering of LDPE samples by absorbance and thickness

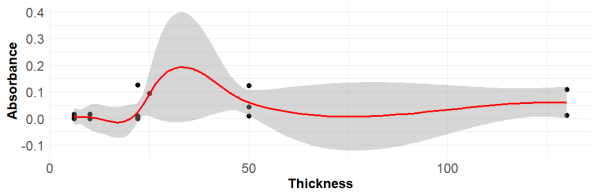

Figure 8. LOESS regression showing non-linear trend between thickness and absorbance

IV. CONCLUSION

This study successfully validated a UV-Vis

spectrophotometric method for determining total UV-absorbing contaminants migrating from LDPE materials into 8% ethanol. The validated extraction method demonstrated reliability in measuring migration levels, with acceptable precision across low to high analyte levels, with established acceptance criteria for routine application.

Profiling of 22 commercially available and locally manufactured LDPE bags revealed that most samples exhibited non-detectable to low levels of migration. Although a few samples showed elevated absorbance values, none exceeded the FDA's regulatory limit, indicating general compliance with food safety standards. Statistical analyses further suggested that while LDPE thickness moderately influences migration, other factors such as formulation and processing play a significant role. Overall, the validated method provides a reliable tool for regulatory screening and quality control of plastic food contact materials, supporting ongoing efforts to ensure consumer safety.

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