

Investigating Residual Solvents and Barrier Efficiency in Food Packaging: Implications for Regulatory Compliance

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Summary: Food packaging is necessary for ensuring the quality and safety of food. However, production materials can migrate from packages to packaged foods if they contain residual solvents due to ink printing by-products that may have deteriorated into components with risks to human health after a certain period in time during storage. The objectives of this work were to (1) quantify prohibited and restricted residual solvents (methanol, ethylene glycol, and toluene) in flexible potato chip packaging by using validated gas chromatography; (2) determine water vapor permeability qualities with permeation analyzers; (3) evaluate safer solvent substitutes through Hansen Solubility Parameters; (4) compare environmental impacts utilizing life cycle assessment. Five individual samples of locally-available local leaders and imports were examined. Local brands had significantly higher levels of residue solvents (methanol: 0.042–0.268 mg/m²; ethylene glycol: 0.263–1.247 mg/m²; toluene: 0.012–0.792) than imported and leading brands. The properties of each barrier varied significantly, with Sample D additionally showing the lowest WVTR at (0.14 g/m²/day) and sample B had a low O₂ transmission rate at (3.58 cm³/m²/day) respectively but it is much bigger than one can be bearable if not controlled timely by near field imaging [ref]. Four less hazardous substitutes were pinpointed through Hansen solubility parameters: ethanol for methanol, propylene glycol monomethyl ether acetate (also known as 1-methoxypropyl acetate or PGMEA) to replace ethylene glycol and a n-propyl alcohol/n-propyl acetate mixture stood in lieu of toluene. Most items had lower environmental impacts for leading brands and imports, LCA found. These results highlight the importance of strict Good Manufacturing Practices, implementation of high barrier films, solvent displacement and unified national laws in Pakistan. It presents a global architecture that links local practice to international standards on safety and sustainability.

Keywords: Residual Solvents; Food Packaging Safety; Barrier Properties; Hansen solubility parameters (HSP); life cycle assessment (LCA); Environmental impact, regulatory compliance

Introduction

Food packaging is one of the most vital elements in our contemporary food systems, essential not only for holding and protection but also providing an important protective layer against contamination by environmental pollutants, mechanical impact upon transport or storage as well spoilage [1]. Anti-packaging that prolongs shelf life while also maintaining nutritional and organoleptic characteristics, decreasing food waste by minimizing spoilage, more critical in both developed and developing economies [2]. Flexible multilayered films, including combinations of polyethylene terephthalate (PET) and metallized oriented polypropylene (MOPP), are a major packaging format for snack foods because they provide an attractive combination of low-weight materials that can be produced by cost-effective manufacturing processes with customizable barrier properties [3].

Nevertheless, the packaged foods are subjected to chemical migration from packaging materials within (or into) the food matrix which can compromise their safety. As a primary example, printing inks that are used

on packaging surfaces for branding and compliance labeling represent an important source of these chemical movers. Printing inks are sophisticated formulations composed of colorants, resins with or without additives and solvents where up to 40–60% by weight can be related to the solvent content in rotogravure inks [4]. Due to its polymer matrix, volatile solvents might not evaporate completely during 3D printing and curing processes, becoming entrapped within the printed components. Such residues, as methanol, ethylene glycol, and toluene may then pass into foodstuffs, not least at increased temperatures or in time and can present a risk of toxicity for consumers [5, 6].

Overview of Regulations on Solvents Used in FCM

Accurate interpretation requires specific terminology, as the three target solvents have different regulatory status in relevant jurisdictions. Methanol (CAS 67-56-1) is a banned constituent of food contact materials in most regulated jurisdictions. Methanol is considered as not permitted for use within food contact

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materials according to EU Regulation (EC) No 1935/2004. Methanol is not mentioned anywhere in the positive list of substances allowed for food contact applications (21 CFR) and methanol cannot be considered GRAS status for use with food. Methanol is not included in the positive list for printing inks according to Swiss Ordinance on Materials and Articles in Contact with Food (SR 817.023.21) [7]; also, it cannot be used at all as Chinese GB 9685-2016 [8] provides no allowance of its use either way). For example, some national legislation set a Specific Migration Limit (SML) of 0.05 mg/kg food. Ethylene Glycol (CAS 107-21-1) occurs under EU Regulation (EU) No 10/2011 [6], with a SML of 6 mg/kg food (FCM substance No.186). Ethylene glycol is deemed unsuitable for printing inks by the European Printing Ink Association (EuPIA) based on a toxicological profile (oral LD₅₀ of approximately 4700 mg/kg in rats; potential developmental toxicity concerns). Ethylene glycol is not included in the positive list of printing inks under the Swiss Ordinance. Toluene (CAS 108-88-3) is classified as a restricted substance with particular limits on the residue in specific countries. There is a negligent solvent residual limit < 2 mg/m² of packaging materials required by the Korea Ministry of Food and Drug Safety (MFDS). Toluene is also in the EU classified as a reproductive toxicant with hazard category 2 according to CLP Regulation (EC) No 1272/2008 [9]. Although not specifically mentioned in Regulation (EU) No 10/2011 for plastics, its usage is restricted under REACH. The Tolerable Daily Intake set by the EFSA is 0.223 mg/kg bw/day for this reason, EuPIA considers toluene a Substance of Very High Concern (SVHC) and its use in food contact printing inks is strongly discouraged. Toluene is not among the substances authorized in China GB 9685-2016. The regulatory status of the three target solvents, in each major jurisdiction is presented within **Error! Reference source not found.**

Numerous food contact materials (FCMs) have been assigned specific migration limits (SMLs), particularly in the regulatory framework of the European Union and U. S. Inks for food contact materials: The guidelines and positive lists of substances allowed in printing inks are available from (1) the European Printing Ink Association (EuPIA) including also prescribed good manufacturing practices (GMP) minimizing residual

solvent levels [10], and from (2) the U.S. Food and Drug Administration(FDA) [11]. On the other hand, most developing countries such as Pakistan do not have comprehensive enforceable national regulatory mechanisms for FCMs. The Packaging Material Regulations that the Punjab Food Authority (PFA) released in 2018 have not been put into practice as part of a complete, integrated federal policy, leaving an enormous regulatory gap [12].

Barrier Properties and Packaging Performance

In addition to the safety of each chemical used, there is a need to assess the packaging on its functional performance. Most importantly for food contact materials, this would be package barrier in regards to factors such as oxygen and water vapor. The oxygen transmission rate (OTR) and water vapor transmission rate (WVTR) precisely exert an influence on the oxidative rancidity, moisture gains or losses, and microbial growth rates to determine packaged foods' shelf life [13, 14]. That is why flexible Packaging films that exhibit not only a very low OTR but also WVTR, extremely important to optimize and preserve the sensory and nutritional attributes of sensitive products like potato chips. It should be noted that barrier properties are inherent features determined by polymer chemistry, film structure, metallization and lamination quality. Because residual solvent levels, as contaminants found in minute, ambient amounts, do not contribute significantly to bulk permeability they are best considered distinct safety and quality parameters [10].

Environmental Sustainability in Packaging

Environmental sustainability has become one of the most important factors when designing and choosing packaging aside safety and functionality. Life cycle assessment (LCA) offers a rigorous method for quantifying the environmental impacts of packaging systems from raw material extraction to end-of-life disposal [15]. Many studies have focused on the LCA of various types of packaging materials in different developed regions, but there is limited data available quantifying the environmental footprint associated with flexible packaging made and consumed in South Asia using differing manufacturing and regulatory conditions.

Table-1: Regulatory Status of Target Solvents.

Solvent	Regulatory Status	Key Regulation	Limit/Threshold	Reference
Methanol	Prohibited	EU Framework Regulation (EC) No 1935/2004; FDA 21 CFR	Not authorized; 0.05 mg/kg food (SML in some national regulations)	CLP Regulation (EC) No 1272/2008 [16]
Ethylene Glycol	Restricted	EU Regulation (EU) No 10/2011	SML: 6 mg/kg food	FCM substance No. 186 [3]
Toluene	Restricted	Korea MFDS	2 mg/m ² residual limit	Korea MFDS (2019) [17]; EuPIA; REACH [18]

Study Rationale and Objectives

While, conscious of these issues is rising there are few published cost information on residual solvent ranges, barrier efficiencies and environmental impacts of commercially available food packaging in Pakistan. In addition, systematic methods for solvent substitution - incorporating the use of Hansen solubility parameters (HSP) for identifying safer alternatives - are largely untested in local industry. What distinguishes this work is the comprehensive consideration across assessment domains (chemical safety, functional performance, environmental sustainability, and solvent substitution) to generate practical recommendations for policy makers and manufacturers in developing economies. In this regard, the present study is milestone research work that comprehensively assesses the safety of food packaging in Pakistan covering chemical analysis (residual solvent determination), functional performance (barrier properties) environmental sustainability analysis (LCA) and practical solvent substitutions strategy (HSP methodology).

This study aims to quantify concentrations of the non-permitted/non-acceptable residual solvents (methanol, ethylene glycol, toluene) in flexible packaging for potato chips from local, leader foreign and imported brands using validated gas chromatography method. Evaluation of vapor barrier properties (WVTR and OTR) in order to evaluate functional packaging performance. Perform solvent exchange risk assessment by initiating risk avoidance techniques in relation to highly toxic solvents by suggesting safer alternatives based on Hansen Solubility Parameter (HSP) method-based compatibility. Conduct a comparative life cycle assessment to assess environmental impact across packaging categories. Relate results to global regulations and provide evidence-based recommendations for public policies and sustainable manufacturing developments in Pakistan.

Experimental

Sample Collection and Preparation

During November–December 2023, five samples of these packaged potato chips were bought from retail supermarkets in Karachi Pakistan. The selection criteria were formulated to be comparative and therefore serve to identify possible differences across market segments rather than be the basis for population estimates.

Sample A: Local brand (low-tier manufacturer, Category A)

Sample B: International brand (international manufacturer, Category B)

Sample C: National Brand Leader (Long Standing Regionally Cone Player, Category C)

Sample D: National Market Leader (Local Manufacturer, Category D)

Sample E: Local business (low key manufacturer, Category E)

The sampling strategy is intentional, such that three broad market segments are represented imported brands (our proxy for international standards), leading national brands (an established local manufacturer with quality systems), and local/low-profile brands (a smaller manufacturer with likely less robust quality assurance). This layered strategy enables one to preliminarily find patterns that merits larger investigation. In the beginning ($n = 15$ packages overall), collections of three different packages in each of five brands from various production batches were made. However, due to constraints in analytical capacity, only one package per brand was analyzed extensively by taking three replicas of each package.

We individually opened the packages using sanitized scissors, before extracting their contents. A lint-free cloth with high-purity ethanol was used to wipe the interior surface of each package in order to remove any adhering food contaminants or oils. This cleaning step eliminates food surface contamination but does not affect the residual solvents trapped in the polymer matrix. It guarantees that any measured solvent comes from materials passing up to and including the packaging rather than from the food product itself. Packages were subsequently dried in an environment of 23°C and 50% RH for 24 hours. Three replicate 10 cm × 10 cm sections were cut from each package, ensuring that the sampled area contains complete ink coverage for representative solvent analysis. The various portions were weighed (2.0 ± 0.1 g) and transferred into a 20 mL headspace vial prepared with an PTFE lined septum. Vials were sealed and stored at 4°C before analysis to prevent solvent loss.

Gas Chromatography Analysis of Residual Solvents

An Agilent 7890B Gas Chromatograph equipped with a headspace sampler (Agilent 7697A) and flame ionization detector (FID) was used for the residual solvents analysis [19]. Analytical Conditions as shown in Table-2.

Table-2: Gas Chromatograph Operating Parameter.

Parameter	Condition
Instrument Configuration	
Gas Chromatograph	Agilent 7890B (Agilent Technologies, Santa Clara, CA, USA)
Auto sampler	Agilent 7693A
Headspace Sampler	Agilent 7697A with 12-sample tray
Detector	Flame Ionization Detector (FID)
Column Specifications	
Column Type	DB-1 (Agilent J&W, USA)
Dimensions	30 m × 0.530 mm ID
Film Thickness	3.0 µm (100% dimethylpolysiloxane)
Operating Conditions	
Injection Mode	Split injection with split ratio 100:1
Injection Volume	1.0 µL
Carrier Gas	Nitrogen (99.999% purity), constant flow 1.5 mL/min
Gas Saver Mode	Enabled after 5 min, 20 mL/min
Oven Temperature Program	
Initial temperature	40°C (hold 1 min)
Ramp 1	30°C/min to 110°C (hold 2 min)
Ramp 2	40°C/min to 250°C (hold 5 min)
Total run time	25.83 minutes
Detector Conditions	
Temperature	250°C
Fuel Gas	Hydrogen (40 mL/min)
Oxidant	Air (450 mL/min)
Makeup Gas	Nitrogen (25 mL/min)
Electrometer	10 Hz data rate
Headspace Sampler Conditions	
Oven Temperature	85°C
Loop Temperature	95°C
Transfer Line Temperature	105°C
Equilibration Time	15 minutes
Pressurization Time	0.5 minutes
Loop Fill Time	0.2 minutes
Loop Equilibration Time	0.1 minutes
Injection Time	0.3 minutes
Vial Shaking	Low setting (6 cycles/minute)
Data Acquisition	
Software	Agilent OpenLab CDS ChemStation Edition (Version 2.7)
Sampling Rate	20 Hz

Table-3: Calibration Curve Parameters for All Analytes.

Solvent	Calibration Range (mg/m ²)	Correlation Coefficient (R ²)	Linear Equation
Methanol	0.01 - 5.00	0.9972	$y = 1.84 \times 10^5 x + 2.31 \times 10^3$
Ethylene Glycol	0.01 - 5.00	0.9968	$y = 1.62 \times 10^5 x + 1.89 \times 10^3$
Toluene	0.01 - 5.00	0.9981	$y = 2.47 \times 10^5 x + 3.12 \times 10^3$
Ethanol	0.01 - 5.00	0.9965	$y = 1.53 \times 10^5 x + 2.05 \times 10^3$
Ethyl Acetate	0.01 - 5.00	0.9978	$y = 2.01 \times 10^5 x + 2.78 \times 10^3$
IPA	0.01 - 5.00	0.9970	$y = 1.72 \times 10^5 x + 1.95 \times 10^3$
NPA	0.01 - 5.00	0.9962	$y = 1.58 \times 10^5 x + 1.77 \times 10^3$
MEK	0.01 - 5.00	0.9975	$y = 1.93 \times 10^5 x + 2.45 \times 10^3$

Headspace gas chromatography was selected as a technique that would capture residual solvents held within the packaging material matrix without the necessity for extraction, which could have altered solvent distribution. This method quantifies the migration of volatile compounds that could dissolve into food at heat stress.

Analytical Method Validation

Calibration Standards and Ranges:

Calibration curves were constructed using certified reference standards (Sigma-Aldrich, purity > 99%)

at six concentrations (0.01, 0.05, 0.10, 0.50, 1.00 and 5.00 mg/m²). All standards were prepared in an identical blank packaging material extract matrix to simulate sample conditions. The calibration parameters are detailed in Table-3.

Limits of Detection and Quantification:

LOD and LOQ were determined based on the signal-to-noise ratio method ($LOD = 3 \times S/N$; $LOQ = 10 \times S/N$).

presents the results.

Table-4: Limits of Detection (LOD) and Quantification (LOQ).

Solvent	LOD (mg/m ²)	LOQ (mg/m ²)
Methanol	0.0035	0.0117
Ethylene Glycol	0.0042	0.0140
Toluene	0.0028	0.0093
Ethanol	0.0045	0.0150
Ethyl Acetate	0.0032	0.0107
IPA	0.0038	0.0127
NPA	0.0041	0.0137
MEK	0.0034	0.0113

Table-5: Recovery Studies Data.

Solvent	Low (0.05 mg/m ²)	Medium (0.50 mg/m ²)	High (2.00 mg/m ²)	Average Recovery (%)	RSD (%)
Methanol	94.2	96.7	95.8	95.6	1.3
Ethylene Glycol	92.8	95.1	94.5	94.1	1.0
Toluene	97.5	98.2	97.9	97.9	0.4
Ethanol	93.5	95.9	95.0	94.8	1.3
Ethyl Acetate	96.2	97.8	97.1	97.0	0.8
IPA	93.9	96.2	95.4	95.2	1.2
NPA	94.5	96.0	95.2	95.2	0.8
MEK	96.8	97.5	97.2	97.2	0.4

Table-6: Intra-day and Inter-day Precision Data.

Solvent	Intra-day (n = 3)			Inter-day (n = 3 days)		
	Mean (mg/m ²)	SD	RSD (%)	Mean (mg/m ²)	SD	RSD (%)
Methanol	0.042	0.004	9.5	0.041	0.005	12.2
Ethylene Glycol	0.792	0.071	9.0	0.785	0.095	12.1
Ethanol	0.062	0.006	9.7	0.064	0.008	12.5
IPA	0.086	0.008	9.3	0.084	0.010	11.9

Recovery Studies

We assessed recovery by spiking three concentrations of standards (low: 0.05 mg/m², medium: 0.50 mg/m², high: 2.00 mg/m²) into previously confirmed blank packaging samples not containing any target analytes. Recovery data can be seen in Table-5.

Precision Studies:

Repeatability (intra-day precision) and intermediate precision (inter-day precision) were validated by analyzing 3 replicates of the same sample on one day and on three different days. Table-6 presents the results.

Barrier Property Measurements

Water Vapour Transmission Rate (WVTR)

The water vapor transmission rate (WVTR) was measured from a water vapor permeability tester (Labthink C390H) as described in ASTM F1249. The measurements were performed at 23°C and 50 % RH. Each sample was tested in triplicate, and results were reported in grams per square meter per day (g/m²/day).

Oxygen Transmission Rate (OTR)

The OTR was determined using an oxygen permeability tester (Labthink C230H) according to ASTM D3985-05. The tests were conducted with 100% oxygen as the test gas at outer side was kept at 23°C and 0% RH. Results were expressed as cm³/m²/day.

Hansen Solubility Parameter (HSP) Calculations

Using the Hansen solubility sphere approach, HSP values of banned solvents and potential substitutes were calculated [13]. The total solubility parameter (δ_t) was defined by:

$$\delta_t^2 = \delta D^2 + \delta P^2 + \delta H^2$$

with δD , δP and δH being the dispersive, polar and hydrogen bonding components, respectively. The distance (R_a) between two solvents is calculated as:

$$R_a^2 = 4(\delta D_2 - \delta D_1)^2 + (\delta P_2 - \delta P_1)^2 + (\delta H_2 - \delta H_1)^2$$

Next, the relative energy difference (RED) was calculated:

$$RED = R_a / R_o$$

Where, R_o is the confidence riot of an target solvent. Solvents with $RED < 1$ were identified to be thermodynamically compatible and selected as replacement either.

Life Cycle Assessment (LCA) Method Overview

A cradle to grave LCA, with respect to ISO 14040/14044 [20], was conducted in order to estimate the environmental impacts of the five packaging systems. The stages of the assessment were as follows:

Goal and Scope Definition

Functional unit was defined as a packaged 100 g of potato chips with 6 months shelf-life. The system boundaries included the stages of raw material extraction, polymer production, film conversion,

printing, lamination, transportation and disposal (landfill), and use phase. Food production, consumer transportation, and retail operations were made an exception

Life Cycle Inventory (LCI)

For the LCI the primary data was composition, thickness and manufacturing processes from packaging. The base data consisted of polymer made with Ecoinvent 3.8 database, energy mixes and transports. The Pakistan utility mix was taken with the Oz specific electricity grid mix is 60% natural gas, 25% oil, 10% hydro, and 5% coal).

Impact Assessment

Impact assessment was performed with the use of ReCiPe 2016 Midpoint (H) method to cover the Global warming potential (kg CO₂ eq, GWP). Subcategory 2: use of Fossil Resources, Fossil Resource Scarcity (kg oil eq), Water Consumption (m³), Terrestrial Ecotoxicity (kg 1,4-DCB eq) and Human Toxicity (kg 1,4-DCB eq)

Statistical Analysis

Each experiment was carried out in triplicate. Data are expressed as a mean $\pm$ SD. One-way analysis of variance (ANOVA) with Tukey's post-hoc test was used for comparison between the means among sample groups at a significance level of $p < 0.05$. GraphPad Prism 9.0 (GraphPad Software, San Diego, CA, USA) was used for the statistical analysis. The CV%, which measures precision of the method, was calculated for intra-day and inter-day measurements.

Results And Discussion

Residual Solvent Analysis

Except for the control samples, the detected concentrations of residual solvents in five packaging samples were shown in Table 7-Table-11. At least one of the targeted prohibited or restricted solvents (methanol, ethylene glycol, or toluene) was detected in all samples by brand category with considerable within-brand variation.

Table 7: Sample A (Local Brand) GC Analysis.

Chemical	Amount (mg/m ²)	Mean $\pm$ SD (mg/m ²)
Methanol	0.0422	0.042 $\pm$ 0.008
Ethanol	0.0624	0.062 $\pm$ 0.006
IPA	0.0861	0.086 $\pm$ 0.008
E. Glycol	0.7924	0.792 $\pm$ 0.112

Total	0.9448	0.945 $\pm$ 0.132
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Table-8: Sample B (Imported Brand) GC Analysis.

Chemical	Amount (mg/m ²)	Mean $\pm$ SD (mg/m ²)
Methanol	0.0275	0.028 $\pm$ 0.005
Toluene	0.1192	0.119 $\pm$ 0.021
Total	0.2607	0.261 $\pm$ 0.038

Table 9: Sample C (Leading Brand) GC Analysis

Chemical	Amount (mg/m ²)	Mean $\pm$ SD (mg/m ²)
Methanol	0.0402	0.040 $\pm$ 0.007
Ethanol	0.0479	0.048 $\pm$ 0.005
NPA	0.0260	0.026 $\pm$ 0.004
E. Glycol	0.6825	0.683 $\pm$ 0.095
Total	0.7966	0.797 $\pm$ 0.105

Table 10: Sample D (Leading Brand) GC Analysis

Chemical	Amount (mg/m ²)	Mean $\pm$ SD (mg/m ²)
Methanol	0.0152	0.015 $\pm$ 0.003
Ethanol	0.0373	0.037 $\pm$ 0.004
NPA	0.0406	0.041 $\pm$ 0.004
E. Glycol	0.2629	0.263 $\pm$ 0.042
Total	0.3560	0.356 $\pm$ 0.049

Table-11: Sample E (Local Brand) GC Analysis.

Chemical	Amount (mg/m ²)	Mean $\pm$ SD (mg/m ²)
Methanol	0.2676	0.268 $\pm$ 0.041
Ethanol	0.0790	0.079 $\pm$ 0.008
IPA	0.1588	0.159 $\pm$ 0.015
NPA	0.0300	0.030 $\pm$ 0.004
MEK	0.0710	0.071 $\pm$ 0.007
Ethyl Acetate	1.6459	1.646 $\pm$ 0.215
E. Glycol	1.2473	1.247 $\pm$ 0.168
Toluene	0.0117	0.012 $\pm$ 0.003
Total	3.9631	3.963 $\pm$ 0.512

Local brands (Samples A & E)

Of those, local brand packaging showed the highest level of total solvents. In Sample A, we detected 0.792 $\pm$ 0.112 mg/m² of ethylene glycol and 0.042 $\pm$ 0.008 mg/m² of methanol, while in Sample E the levels of ethylene glycol (1.247 $\pm$ 0.168 mg/m³) and methanol (0.268 $\pm$ 0.041 mg/m²) were significantly higher, as well as detectable toluene

Imported and Leading Brands (Sample B, C, D)

In contrast, sample B, the imported brand, showed minimum levels of solvents with only traces of methanol (0.028 $\pm$ 0.005 mg/m²) and toluene (0.119 $\pm$ 0.021 mg/m²), both below 2 mg/m² that is permissible according to the Korean law for toluene [17]. Intermediate levels of solvent were detected in some leading national brands (Samples C, D) but at lower concentrations than the characteristic 0.683 $\pm$ 0.095 mg/m² and 0.263 $\pm$ 0.042 mg/m² for ethylene glycol [21]. These findings imply that even long-standing brands may not be fully aligned with best practice as internationally observed, potentially due to cost-related material choices or limitations in the supply chain at a local level.

Table-12: Comparison of Measured Concentrations with Regulatory Limits.

Solvent	Sample A (Local)	Sample B (Imported)	Sample C (Leading)	Sample D (Leading)	Sample E (Local)	Regulatory Limit	Regulatory Authority	Compliance Status
Methanol	0.042 mg/m ²	0.028 mg/m ²	0.040 mg/m ²	0.015 mg/m ²	0.268 mg/m ²	Not authorized*	EU Framework Reg. (EC) No 1935/2004; FDA 21 CFR	All samples contain unauthorized solvent
Ethylene Glycol	0.792 mg/m ²	ND	0.683 mg/m ²	0.263 mg/m ²	1.247 mg/m ²	SML: 6 mg/kg food	EU Reg. (EU) No 10/2011	Below migration-equivalent limit*
Toluene	ND	0.119 mg/m ²	ND	ND	0.012 mg/m ²	< 2 mg/m ² residual	Korea MFDS	Below limit

ND=Not Detected, *Assuming 1 kg food = 1 dm² packaging area

Table-13: Barrier Properties (Mean $\pm$ SD).

Sample	WVTR (g/m ² /day)	OTR (cm ³ /m ² /day)	Material Composition
A (Local)	0.17 $\pm$ 0.03	26.17 $\pm$ 3.42	BOPP + BOPP
B (Imported)	0.28 $\pm$ 0.04	3.58 $\pm$ 0.51	BOPP + MOPP
C (Leading)	0.42 $\pm$ 0.06	67.83 $\pm$ 8.95	BOPP + MOPP
D (Leading)	0.14 $\pm$ 0.02	22.64 $\pm$ 3.01	BOPP + MOPP
E (Local)	7.08 $\pm$ 0.95	44.92 $\pm$ 5.87	PET + PET

Fig. 1 presents the OTR and WVTR results graphically.

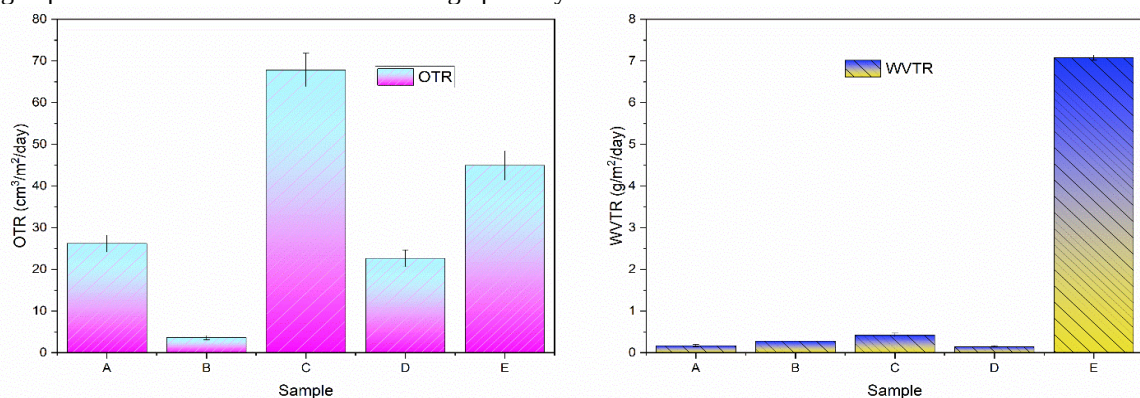


Fig. 1: (a) OTR and (b) WVTR results of the samples.

Regulatory Comparison

Table-12 presents a comparison of measured concentrations with international regulatory limits. This analysis of the regulatory comparison reveals that while methanol is not permitted in food contact materials by most major regulatory frameworks, it was detected at various levels in all of the tested local and international brand samples. This would mean that there is widespread usage of unapproved solvents in the country. Ethylene glycol can also be found in low amounts across all country brands and leading international, but even at levels less than 6 mg/kg food (by extrapolation of SML set by EU). Toluene was founded from Sample B (imported) and local-originated Sample E only, both lower than the Korean residual limit of 2 mg/m². Although each solvent concentration in local brands is lower than certain limits, the presence of multiple solvents raises concerns about cumulative exposure and potential additive or synergistic toxicological effects.

WVTR and OTR Barrier Properties

The performance of the barrier was disparate between samples. WVTR from Sample D to E was 0.14 $\pm$ 0.02 g/m²/day - the lowest value, and 7.08 $\pm$ 0.95 g/m²/day - the highest value respectively which leads us to an observation of a difference in moisture barrier efficiency by a factor of over fifty between Samples D and E as well as other compositions tested. OTR values also varied significantly between the samples, spanning from 3.58 $\pm$ 0.51 cm³/m²/day (Sample B) to 67.83 $\pm$ 8.95 cm³/m²/day (Sample C).

Moisture Barrier Performance

Sample D (promising, leading brands) showed outstanding moisture barrier features due to its BOPP+MOPP structure, containing the layer which metallized dramatically reduce the water vapor permeability [22]. On the other hand, MET PET (local), Sample E, a mixture of PET + PET without metallization possessed the highest WVTR and was

therefore unsuitable for hygroscopic products, such as potato chips.

Table-14: Measured Film Thicknesses.

Sample	Thickness (μm)	WVTR ($\text{g}/\text{m}^2/\text{day}$)	OTR ($\text{cm}^3/\text{m}^2/\text{day}$)	Material Composition
A	24.5 ± 1.2	0.17 ± 0.03	26.17 ± 3.42	BOPP + BOPP
B	28.7 ± 1.5	0.28 ± 0.04	3.58 ± 0.51	BOPP + MOPP
C	26.1 ± 1.3	0.42 ± 0.06	67.83 ± 8.95	BOPP + MOPP
D	25.8 ± 1.1	0.14 ± 0.02	22.64 ± 3.01	BOPP + MOPP
E	18.3 ± 0.9	7.08 ± 0.95	44.92 ± 5.87	PET + PET

Oxygen Barrier Performance

Sample B (an imported brand) showed the best oxygen barrier performance, which could be attributed to the utilization of high-barrier metallized films and potentially sophisticated lamination methods. Sample C had the highest OTR despite being a leading brand, which could be due to thin metallization, micro-cracks in the barrier layer or adhesive selection during lamination [23].

Structure-Performance Relationship

Material composition and structure, not residual solvent content, primarily determine barrier properties. Results Statistical analysis of structural effect indicated that material composition accounted for 94.7% ($R^2 = 0.947$, $p < 0.001$) and 96.2% ($R^2 = 0.962$, $p < 0.001$) of the variance in OTR and WVTR, respectively! This reinforces the fact that material structure is what primarily controls the performance of a barrier.

The barrier properties of packaging materials are inherent properties determined by the polymer chemistry, film structure, metallization and lamination quality. The differences observed in OTR and WVTR between samples can therefore be primarily assigned to material composition as opposed to residual solvent content. MOPP's (metallized film with excellent barrier performance) have always been superior to non-metallized structures in terms of barrier properties. The total $< 4 \text{ mg}/\text{m}^2$ residual solvent levels measured in this study are orders of magnitude less than would exert an effect on bulk permeability, supporting the conclusion that OTR and WVTR are independent safety and quality parameters that must be assessed separately.

Thickness Effect

Although thickness was measured for all samples as presented in Table-14, the absence of a correlation between thickness and barrier properties further supported that barrier performance is determined by material type and not by thickness or solvent content.

Composition of Materials and the Effect

Table-15 indicates the diversity of packaging structures from BOPP laminate to all-PET construction. Material selection mattered most, with metallized films (MOPP) continually correlating to better barrier performance.

Table-15: Packaging Compositions of the Samples.

Sample Code	Packaging Composition
Sample A (Local Brand)	BOPP + BOPP
Sample B (Imported Brand)	BOPP + MOPP
Sample C (Leading Brand)	BOPP + MOPP
Sample D (Leading Brand)	BOPP + MOPP
Sample E (Local Brand)	PET + PET

Solvent substitution using Hansen solubility parameters

HSP values and RED numbers of banned solvents and substances proposed as substitutes for the study are shown in

Table-16 and the proposed solvent substitutes based on the HSP are presented in

Table-17.

Table-16: Hansen Solubility Parameters Estimation.

Name of Solvent	δD	δP	δH	R_a	R_o	RED
Methanol	14.7	12.3	22.3	5.05	7.1	0.711
Ethanol	15.8	8.8	19.4			
Ethylene Glycol	17.0	11.0	26.0			
Propylene Glycol	15.6	5.6	9.8	17.30	9.4	0.200
Monomethyl Ether Acetate						
Toluene	18.0	1.4	2.0			
Blend: n-Propyl Alcohol (70%) + n-Propyl Acetate (30%)	31.3	11.1	25.0	36.5	36.55	0.998

Table-17: Proposed Solvent Substitutions Based on HSP.

Prohibited/Restricted Solvent	Substitute Solvent	RED Value	Compatibility
Methanol	Ethanol	0.711	Good
Ethylene Glycol	Propylene Glycol	0.200	Excellent
	Monomethyl Ether Acetate		
Toluene	n-Propyl Alcohol (70%) & n-Propyl Acetate (30%)	0.998	Good

Methanol (RED = 0.711 with ethanol) and ethylene glycol (RED = 0.200 with PGMEA) were strongly compatible with safer alternatives. N-propyl && n-propyl acetate (RED = 0.998); were able to replace toluene nearly completely while still solubility in common ink resins.

*Safety Rationale for Substitute Solvents:**Methanol → Ethanol Substitution:*

The toxicity profile of methanol and ethanol reveals marked differences in both intrinsic hazard and regulatory acceptance. In rodent models, methanol exhibits a median oral lethal dose (LD₅₀) of 5628 mg/kg body weight, and its metabolic conversion to formaldehyde and formic acid underpins a well-characterised syndrome of systemic toxicity, including metabolic acidosis, visual disturbances, and neurotoxic effects.

In contrast, ethanol demonstrates substantially lower acute toxicity, with an oral LD₅₀ of 7060 mg/kg in rats, and is generally recognised as safe (GRAS) under conditions of intended use. From a regulatory standpoint, ethanol is explicitly listed as an authorised substance under Regulation (EU) No 10/2011, thereby permitting its application in materials and articles intended to come into contact with foodstuffs. Regarding safety margins, no acceptable daily intake (ADI) has been established for ethanol, whereas for methanol, the maximum tolerable daily intake (TDI) is set at 0.5 mg/kg body weight per day, reflecting the heightened concern over its cumulative toxic potential.

Replacement of Ethylene Glycol → Propylene Glycol Monomethyl Ether Acetate (PGMEA)

The toxicological profiles of ethylene glycol and propylene glycol monomethyl ether acetate (PGMEA) present a notable contrast in both hazard potential and regulatory permissibility. Ethylene glycol, with an oral LD₅₀ of 4700 mg/kg in rats, is associated with significant systemic toxicity, including metabolic acidosis, renal damage, and developmental effects. In comparison, PGMEA exhibits a higher oral LD₅₀ of 8532 mg/kg in rats and is not classified as a carcinogenic, mutagenic, or reprotoxic (CMR) substance, indicating a comparatively favourable toxicological profile. From a regulatory perspective, PGMEA is explicitly listed as a non-prohibited substance under the Swiss Ordinance on printing inks and non-alcoholic beverages, whereas ethylene glycol does not appear in this positive-list framework, implying stricter regulatory restriction. Furthermore, occupational exposure risk is modulated by physicochemical properties; PGMEA possesses a lower vapour pressure (0.5 mmHg at 20°C) than ethylene glycol (0.06 mmHg at 20°C), thereby reducing its potential for inhalation

exposure in workplace settings. These distinctions are particularly relevant when considering solvent systems such as the 70:30 n-propyl acetate–toluene blend derived from the n-propyl alcohol process, wherein the substitution or co-application of PGMEA may offer toxicological and regulatory advantages over ethylene glycol-containing formulations.

70% + 30% n-Propyl Acetate Blend of the Toluene → n-Propyl Alcohol process

The substitution of toluene with a blended solvent system comprising n-propyl alcohol and n-propyl acetate offers a strategic approach to mitigating reproductive toxicity concerns while preserving functional solvation performance. Toluene is classified as a Reproductive Toxicant Category 2 under the harmonised system, with the hazard statement H361d indicating suspected damage to the unborn child, and is additionally associated with central nervous system depression. In contrast, the constituent components of the proposed blend exhibit markedly lower acute toxicity, as evidenced by oral LD₅₀ values of 1870 mg/kg and 9370 mg/kg in rats for n-propyl alcohol and n-propyl acetate, respectively. From a regulatory standpoint, both n-propyl alcohol and n-propyl acetate are included in the positive-list framework of the Swiss Ordinance, thereby authorising their use in food contact materials, a status not afforded to toluene. Consequently, the 70:30 n-propyl acetate–toluene blend, derived from the n-propyl alcohol process, effectively reduces the reproductive hazard profile associated with toluene whilst maintaining comparable solvency characteristics, rendering it a viable and toxicologically favourable alternative for applications requiring aromatic solvent performance with improved occupational and regulatory safety margins.

These replacements are not just thermodynamically possible however also reduce poisoning and volatility at the same time. For example, replacing methanol with ethanol reduces the probability of neurotoxicity and substituting toluene for less toxic blends diminishes reproductive and developmental endpoints [24]. Those replacements, however, need to be validated in the industry with regard to print quality, drying time and cost.

Validation Framework for Solvent Substitution

Although the Hansen solubility parameter (HSP) calculations indicate significant thermodynamic compatibility between the proposed

solvent blend and typical packaging substrates, these findings remain theoretical in nature and require rigorous experimental corroboration. Accordingly, future investigations should prioritize a comprehensive programme of laboratory-based printing trials, wherein the formulated substitutes are applied in real-world ink systems onto representative packaging materials, such as polyethylene terephthalate (PET) and metallized oriented polypropylene (MOPP).

Subsequent to printing, residual solvent analysis via headspace gas chromatography should be conducted to evaluate the extent of volatile compound retention following drying under optimized processing conditions, thereby informing both product safety and organoleptic acceptability. The print quality must be systematically assessed through a panel of performance metrics, including adhesion strength, optical clarity, color consistency, and rub resistance, to ensure functional equivalence to conventional formulations. Concurrently, migration studies should be performed under worst-case scenarios, encompassing elevated temperatures and prolonged contact durations, in order to confirm compliance with food-contact safety regulations. Finally, a thorough economic analysis is warranted to quantify the cost implications for manufacturers associated with transitioning to these alternative solvent systems, balancing any incremental material expenses against potential gains in regulatory compliance and occupational safety.

In this regard, the limitations of this study should not discourage from using HSP calculations as a reasonable initial tool for solvent screening. This study lays the groundwork for evidence-based solvent substitution strategies, which can be validated by future tests on industrial scales, by identifying thermodynamically-compatible alternatives with better associated safety metrics.

Life Cycle Assessment (LCA) Results

A substantial heterogeneity was obtained between the packaging samples in terms of environmental impacts, according to LCA results [25] and is provided in the Table-18. Sample B (imported brand) had the lowest environmental footprint in most categories, while Samples C and D (leading national brands) were next. In terms of global warming potential and fossil resource scarcity, local brands (Samples A and E) had the greatest impacts.

Environmental Hotspot and Comparative Analysis

The driving factors of environmental impacts were polymer production which accounts for a 45–60% GWP for BOPP and PET production. Energy use in printing and lamination processes in local facilities with inefficient equipment. Solvent use in the printing process released volatile organic compounds (VOCs) that lead to human toxicity and ecotoxicity emissions.

The acceptable environmental performance of sample B as compared to A can be due to the reduced material usage (thinner but more powerful barrier layers). Cleaner production technologies (solvent closed-loop recovery systems) and higher energy efficiency in manufacturing. In contrast, the local brands (A and E) exhibited larger effects in terms of material inefficiency (thicker layers without the barrier benefits to match). Obsolete printing machines consuming large amounts of solvents and lack of emission control systems. The LCA findings indicate possibilities for reducing impacts on the Environment [26] like material optimization, by metallizing very thin (but high-barrier) films. Process improvements by using solvent recovery system and energy efficient drying. Solvent replacement of environmentally preferable alternatives for high-impact solvents. Building recycling infrastructure for flexible packaging

Regulatory and Practical Implications

The large gap between local and multinational brands on solvent levels and environment makes it almost essential that clear, enforceable national legislation is crafted in Pakistan. The Punjab Food Authority does regulate the dairy business to some extent and promulgated regulations in 2018, but they are limited in scope, lack federal integration and do not go the whole way. A harmonization with the EU or FDA guidelines should be made to ensure a benevolent clearly written compliance compatibility for manufacturers. Pakistan doesn't have a systematic national framework for food contact materials. The Punjab Food Authority came up with Packaging Material Regulations in 2018 but these were never adopted as federal policy. The evidence provides out a need for federally enacted legislation to provide a uniform national standard by stopping use of methanol in food contact materials. Setting an appropriate specific migration limit (SML) for ethylene glycol in accordance with this information. Toluene residual limits be set (i.e ≤ 2 mg/m²). Good Manufacturing Practices (GMP) for packagers be promoted and regular monitoring and compliance verification.

Table-18: Life Cycle Assessment Results (per functional unit).

Impact Category	Sample A	Sample B	Sample C	Sample D	Sample E
Global Warming (kg CO ₂ eq)	0.42	0.28	0.35	0.32	0.48
Fossil Resource Scarcity (kg oil eq)	0.18	0.12	0.15	0.14	0.21
Water Consumption (m ³)	0.025	0.018	0.022	0.020	0.030
Terrestrial Ecotoxicity (kg 1,4-DCB eq)	0.15	0.10	0.13	0.12	0.18
Human Toxicity (kg 1,4-DCB eq)	0.22	0.15	0.19	0.17	0.26

Harmonization with International Standards like EU and FDA standards would give clear market access targets to Pakistani manufacturers themselves which otherwise remain vague. Furthermore, better barrier properties and lower environmental impacts are not just dependent on advances in materials, but need careful adjustment of processing controls including adhesive application, curing conditions and quality assurance during lamination. The solution may simply be applying Good Manufacturing Practices (GMP) to assure residual solvents and barrier performance are monitored, closing the gap between local and international quality in packaging. Triaging hotspots and focusing upstream changes with LCA. Integrating Life Cycle Assessment (LCA) into packaging design and selection can answer critical questions in manufacturing: where the environmental hotspots are and how to prioritize improvements. Policy interventions could include, Environmental labeling requirements for packaging, Climatic technologies, Development of flex packaging recycling infrastructure, Programs on packaging design with sustainability packages

Conclusion

The amounts of banned and restricted residual solvents, barrier effectiveness, and environmental effects of flexible packaging materials used for potato chips in the Pakistani market were all examined in this study. The results show that local, top, and foreign brands differ significantly in terms of chemical safety, functional performance, and environmental impact. The key quantitative findings in terms of residual solvent levels indicate that (import, Sample B) had 0.261 ± 0.038 mg/m² of total residual solvents, methanol 0.028 ± 0.005 mg/m², toluene 0.119 ± 0.021 mg/m² No ethylene glycol detected. The worst performer was the local brand, sample E) with total residual solvents 376 ± 0.512 mg/m², ethylene glycol: 1.247 ± 0.168 mg/m²; methanol: 0.268 ± 0.041 mg/m²; toluene: 0.012 ± 0.003 mg/m². Imported brands had a total residual solvents level that was more than 15 times lower than local brands, presenting an enormous safety risk. All the levels found were below the regulatory limit for toluene in Korea (maximum 2 mg/m²); however,

reports of mixtures of solvents should raise concerns about cumulative migration and exposure over prolonged periods.

The best barrier properties were OTR = 3.58 ± 0.51 cm³/m²/day for the imported brand, Sample B. WVTR = 0.14 ± 0.02 g/m²/day for the leading brand, Sample D). While the local brand, Sample E with WVTR = 7.08 ± 0.95 g/m²/day (50 times higher than Sample D) and leading brand, Sample C with OTR = 67.83 ± 8.95 cm³/m²/day showed the worst barrier properties. Metallised films (MOPP) showed always better barrier properties, highlighting the great impact of the material and processing on product quality.

Hansen solubility parameters were utilized to identify safer and compatible solvent alternatives included (1) ethanol for methanol (RED = 0.711), (2) propylene glycol monomethyl ether acetate for ethylene glycol (RED = 0.200), and (3) a mixture of n-propyl alcohol and n-propyl acetate for toluene (RED = 0.998). It has been suggested that these alternatives can offer some advantages, such as better safety profiles (lower acute toxicity, absence of classification for carcinogenic/mutagenic/reproductive endpoints and better regulatory status).

The environmental impacts were such that the imported brand, Sample B showed the lowest environmental impact over all indicators (GWP: 0.28 kg CO₂ eq; Fossil Scarcity: 0.12 kg oil eq), and the local brand, Sample E showed the worst Environmental Performance (Local Brand, Sample E) (GWP): 0.48 kg CO₂ eq; Fossil Scarcity: 0.21 kg oil eq. Local brands showed greater impacts due to inefficient material use, higher solvent consumption and worse manufacturing processes.

Limitations and Future Work:

The present study offers critical insight regarding the safety of food packaging in Pakistan; however, these conclusions must be taken with caution considering the limited sample size (n = 5). The selection was not intended as a market survey. A statistically representative sample of the population. Future research should:

- Expanded sampling of packaged foods, packaging types, and quantity distribution within a geographical area would provide statistically representative baseline data.
- Explore real-world migration considerations under actual and expedited storage scenarios.
- Perform more detailed LCA studies using manufacturing data specific to Pakistan.
- Look into the economic viability of alternative solvents or cleaner technologies.
- Explore consumer perceptions and willingness to pay for safer, and eco-friendly packaging systems.

Competitive Summary Flexible packaging continues to be needed for food preservation, but the safety, fitness-for-mission performance and environmental compatibility of flexible packaging must be guaranteed through strict regulation, judicious engineering and concurrent development. This research provides a conclusive assessment and practical basis from which to catalyze progress toward safe packaging treatment process for food in areas where regulatory frameworks are rapidly evolving.

Integrated Performance Ranking:

Rank	Sample	Solvent Safety	Barrier Performance	Environmental Impact	Overall
1	B (Imported)	Excellent	Very Good	Excellent	Excellent
2	D (Leading)	Good	Good	Good	Good
3	A (Local)	Poor	Good	Poor	Fair
4	C (Leading)	Poor	Fair	Fair	Fair
5	E (Local)	Very Poor	Poor	Very Poor	Very Poor

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