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FREQUENCY OF PVC TOYS IN NORTHERN ALGERIA AND COMPARISON OF ANALYSIS METHODS: FLAME TEST *VS.* IR SPECTROSCOPY

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Abstract

Introduction. Children's toys are predominantly made from plastic, primarily due to its low cost and durability. Among the polymers used, polyvinyl chloride (PVC) is particularly widespread in toy manufacturing. However, growing concerns about the health and environmental impacts of these materials, especially PVC, necessitate an evaluation of their frequency of use and composition. The main objective of this study is to determine the relative frequency of PVC toys intended for children under 36 months in various regions of northern Algeria, including Setif, Constantine, Annaba, Bejaia, Algiers, and Oran. In parallel, this research aims to compare two analytical methods commonly used to identify PVC in toys: the flame test and infrared (IR) spectroscopy, to determine the most efficient method for rapid and accurate detection.

Methods. This cross-sectional study involved 271 new plastic toys purchased from the six mentioned cities. Among the 152 toys that tested positive in the flame test, 149 (98%) also yielded a positive result in IR spectroscopy. The relative frequency of soft PVC toys was established at 55%, confirmed by IR analysis, while the specificity of the flame test was estimated at 97.6%.

Results. This study highlights a significant presence of PVC in marketed toys, raising concerns about potential health risks for children, particularly due to the presence of toxic plasticizers often associated with this polymer. The comparison between the identification methods showed that while IR spectroscopy is more specific and reliable, the flame test offers notable advantages in terms of speed and low cost, making it an effective tool for initial screening.

Conclusion. It is crucial to strengthen the monitoring of these products and promote the use of safer materials in their production. Further research is needed to assess the chemical composition of plastic toys and their long-term health effects on children, in order to guide toy regulation and safety policies.

Keywords: Flame test, Infrared spectroscopy, Material identification, PVC, frequency, Toys

Introduction

Children's toys are predominantly made from plastic, primarily due to its low cost and high durability [1]. Over the years, global plastic production has significantly increased, reaching 335 million tons in 2016 [2]. Among the various polymers used in toy manufacturing, polyvinyl chloride (PVC) plays a major role due to its plastic properties and widespread use in this sector [3]. However, growing concerns about the potential health and environmental impacts of plastic materials, especially PVC, necessitate an evaluation of their frequency of use and composition.

In this context, the primary objective of this study is to determine the relative frequency of PVC toys intended for children under 36 months, sold in different regions of northern Algeria, including Setif, Constantine, Annaba, Bejaïa, Algiers, and Oran.
Additionally, this research aims to compare two analytical methods commonly used to identify PVC materials in toys: the flame test and infrared (IR) spectroscopy, to determine the most efficient method for rapid and accurate detection.

Materials and Methods

I.1. Study Area

We selected six cities in the northern region of Algeria for the study: Setif, Constantine, Annaba, Bejaïa, Algiers, and Oran (Figure 1).

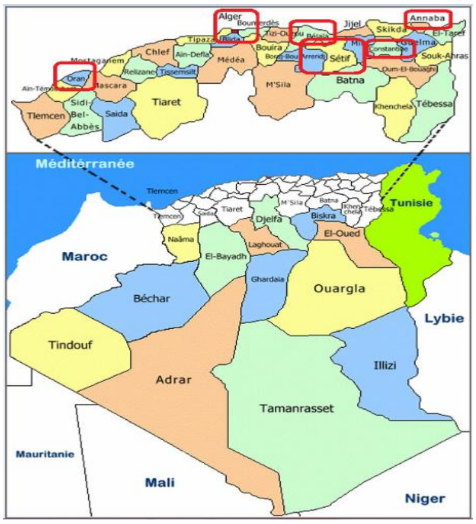

Figure 1. Presentation of the Study Area.

I.2. Type of Study

This is a cross-sectional study focusing on determining the frequency of PVC toys marketed in northern Algeria, using two analytical methods (Flame Test *vs*. IR Spectroscopy).

I.3.Sampling

We opted for 271 new plastic toys purchased from the aforementioned six cities.

I.4. Identification of the Chemical Nature of the Polymer

I.4.1.Principle

The chemical nature of the polymer in the selected toys was identified using the *Beilstein* test (also known as the flame test) and confirmed by infrared (IR) spectroscopy. The flame test is a non-specific method that detects chloride ions in products. The presence of these ions helps exclude toys that are not PVC [4]. All toys included in our study underwent the flame test. IR spectroscopy serves as a confirmation method used to validate the positive results obtained from the flame test.

a.Flame Test (*Beilstein* Test)

Heating a sample of the polymer can provide several preliminary approaches for analysis. Flame tests can differentiate polymers since the flame produced by a burning polymer exhibits characteristic differences depending on the material's structure [4]. The *Beilstein* test has been used for decades in the analysis of organic and polymeric materials and is recommended for detecting PVC in products [5]. This test relies on the reaction of chlorine in the presence of copper compounds at high temperatures, such as those produced by burner flames. This reaction results in excited copper atoms or ions (copper in the presence of chloride ions produces a bright green color), giving the normally colorless flame a bright green (or sometimes blue-green) [4-7].

b. IR Analysis

The chemical nature of the PVC polymer was confirmed by IR spectroscopy. IR analysis is a popular method for characterizing polymers. It is a vibrational technique in which absorption bands at specific wavelengths allow for the identification of specific functional groups in the polymer structure [4, 8]. For soft PVC measurements, peaks around 1425 cm^{-1}, 959 cm^{-1}, and 610 cm^{-1} are specific to PVC. In some cases, verification is performed using the C-Cl stretching vibration near 610 cm^{-1} [9]. Figure 2 shows the

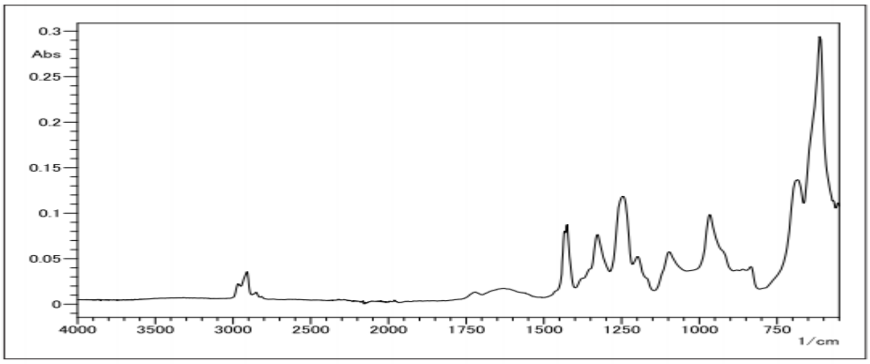

Figure 2. Specific IR Spectrum of PVC [4].

I.4.2. Reagents, Materials, and Equipment

a. Reagents

- Hydrochloric acid HCl (Sigma-Aldrich, 07102-2.5L-GL, lot # SZBE1990V).
- Nitric acid HNO_3 (Fluka, 30709-2.5L-GL, lot # I345S).
- PVC powder (Sigma-Aldrich Chemie GmbH, CAS Number: 9002-86-2, 81388-250G).

b. Materials

- Bunsen burner.
- Copper wire.
- Scalpel.

c. IR Equipment

c1. Fourier Transform Infrared (FTIR) Spectrometer (8400S - SHIMADZU)

This method is based on the interference of radiation between two beams to produce an interferogram, which is a signal generated based on variations in the path length between the two beams. The domains of distance and

frequency are interconvertible using the mathematical method of Fourier transformation [10]. Fourier transformation is a decoding procedure by which the signal is converted from the time domain, where the signal is a function of delay, to the frequency domain, where it is a function of frequency [4].
specific IR spectrum of PVC

c2. Attenuated Total Reflectance (ATR) IR Spectrometer (IRAffinity - 1S-SHIMADZU)
The ATR technique requires virtually no sample preparation. It allows for rapid and robust measurements of solid and liquid samples, including pastes and difficult-to-handle samples [11]. In our practice, IR-ATR was used to analyze samples with dense coloration, where the IR-FTIR spectrum was challenging to interpret or where obtaining a thin, homogeneous, and transparent film was difficult.

I.4.3. Experimental Part

a. Flame Test

- In the flame of a Bunsen burner, heat a copper wire large enough not to melt too quickly until all colors (except for the slightly bluish and barely visible flame) have disappeared. The flame should show no green coloration;
- Occasionally dipping the wire into a solution of water and 10% nitric acid generally removes unwanted coloring elements. If this operation is unsuccessful, use another wire;
- Once the wire is cleaned, avoid touching it with fingers and ensure the only object it contacts is the material being tested;
- The test should only take place when the flame is perfectly colorless. It is advisable to conduct the test under reduced lighting to better distinguish the flame's color;
- After heating the copper wire to red, quickly bring it into contact with a small piece of plastic toy, then return the wire immediately to the flame. If the flame turns green (or sometimes blue-green), it indicates that the tested toy contains chlorine [4-7].

b. IR Analysis

b1. Preparation of the Blank for IR-FTIR

- FTIR analysis requires the preparation of a potassium bromide (KBr) tablet that is transparent in IR;
- The KBr crystals must be dried in an oven at 60°C and then ground to obtain a very fine and homogeneous powder;
- We prepared the tablet by uniformly filling the mold. The tablet must be transparent and uniform;
- We applied a pressure of 80 bar using a hydraulic press;
- We retrieved the tablet and started the IR-FTIR analysis.

b2. Preparation of Standard PVC

- We ground the standard PVC powder with KBr to dilute it to 1/10;
- We followed the same steps to prepare the transparent and uniform tablets.

b3. Preparation and Analysis of Plastic Toys

There are several methods to examine polymer samples. If the polymer is a thermoplastic, it can be softened by heating and pressed into a thin film using a hydraulic press. Alternatively, the polymer can be dissolved in a volatile solvent, allowing the solution to evaporate into a thin film on an alkali halide plate. Some polymers, such as cross-linked synthetic rubbers, can be microtomed [4].

I.4.4. Calculation of Results

Table 1. Characteristics of Analyzed Toys.

		Number of Items	Percentage
Country of Origin	China	198	73.1%
	Algeria	9	3.3%
	Not Mentioned	64	23.6%
	Total	**271**	**100.0%**
Type of Toys	Animals	53	19.56%
	Dolls	36	13.28%
	Character Miniatures	2	0.74%
	Balloons	23	8.49%
	Bath Toys	49	18.08%
	Balls	11	4.06%
	Blocks	3	1.11%
	Doll Accessories	46	16.97%
	Construction Toys	20	7.38%
	Plastic Food	28	10.33%
	Total	**271**	**100.0%**
Purchase price	<200DA	36	13,3%
	200-400DA	91	33,6%
	400-600DA	93	34,3%
	600-800DA	19	7,0%
	>800DA	32	11,8%
	Total	**271**	**100,0%**

We calculated the relative frequency of PVC toys in relation to the total number of toys analyzed (n=271). The relative frequency is the ratio of the count of a particular modality of the studied variable to the total count of the population, or more generally, to the sample on which this variable is measured [12]. The specificity of the flame test was calculated using the following equation:

Specificity=VN/(VN+FP) **[13].**

where *VN* = True Negatives, and *FP* = False Positives.

RESULTS

II.1. Characteristics of the Studied Population

Our non-random selection consisted of 271 new soft plastic toys purchased from major toy stores located in six cities of northern Algeria (Table 1).

II.2. Identification of the Chemical Nature of the Polymer

Among the 152 toys that showed a positive result in the flame test, 149 of them, or 98%, also yielded a positive result in the IR analysis, while three toys, or 2%, showed a negative result by IR (Table 2).

Table 2. Distribution of Selected Toys According to the Results of the Flame Test (n=271) and IR Analysis (n=152).

		Number	**Percentage**
Flame Test (n=271)	Negative	119	43.9%
	Positive	152	56.1%
	Total	**271**	**100.0%**
IR Analysis (n=152)	Negative	3	2%
	Positive	149	98%
	Total	**152**	**100.0%**

Figure 3 and figure 4 represent the IR spectra of toys S_{33} and A_{40}, obtained using an IR-ATR spectrophotometer.

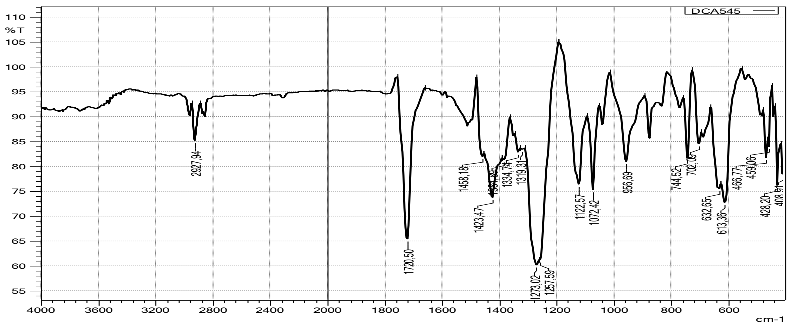

Figure 3. Positive IR-ATR Spectrum of Sample S_{33}**.**

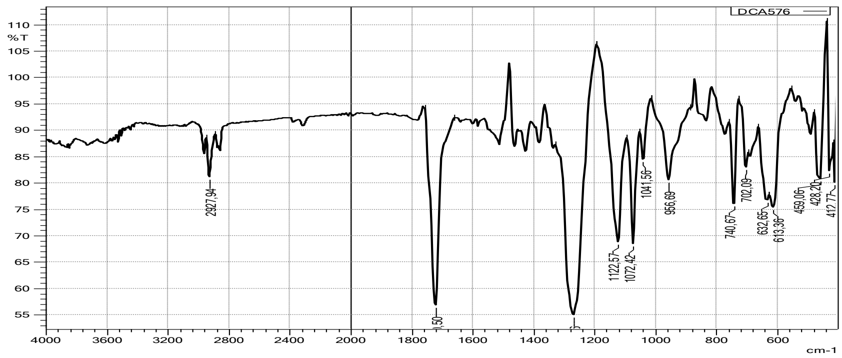

Figure 4. Positive IR-ATR Spectrum of Sample A_{40}.

Figure 5 illustrates the IR spectrum of the standard PVC analyzed by IR-FTIR spectroscopy.

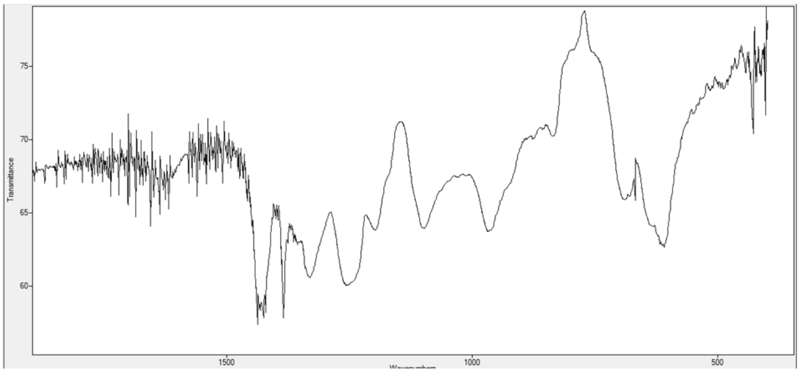

Figure 5. IR Spectrum of Standard PVC Analyzed by IR-FTIR Spectroscopy**.**

II.2.3. Relative Frequency of Soft PVC Toys Marketed in Northern Algeria

The examination by IR spectroscopy confirmed the presence of PVC in 149 of the 271 plastic toys included in our study. The relative frequency of soft PVC toys was established at 55%.

II.2.4. Characteristics of PVC Toys Marketed in Northern Algeria
a. Distribution of PVC Toys by Country of Origin

Among all toys identified as PVC, it was observed that nearly a quarter, or 21.5% (32 toys), came from an unknown source, while 117 toys, or 78.5%, were of Chinese origin.
Among the initially selected 271 toys, 59.1% of the toys from China were PVC, while 40.9% were not PVC. Regarding toys without a specified country of origin, 50% were PVC, and all locally produced toys were not PVC (Table 3).

Table 3. Distribution of Selected Toys by Country of Origin and Polymer Nature (n=271).

	Non PVC	PVC	Total	*p*
Algeria	9	0	9	0,001*
China	81	117	198	
Not Specified	32	32	64	
Total	**122**	**149**	**271**	

There was a statistically significant correlation between the nature of the polymer in toys and their country of origin (p = 0.001).
b. Distribution of PVC toys by toy type
Table 4 represents the distribution of soft PVC toys according to the type of toy.

Table 4. Distribution of soft PVC toys by toy type (n=149).

Type of Toys	Number of Items	Percentage
Animals	37	24.8%
Dolls	33	22.1%
Character Miniatures	2	1.3%
Balloons	16	10.7%
Bath Toys	48	32.2%
Balls	7	4.7%
Blocks	2	1.3%
Doll Accessories	4	2.7%
Total	**149**	**100.0%**

c. Distribution of soft PVC toys by purchase price

Table 5 illustrates the distribution of soft PVC toys according to the purchase price (n=149). More than half of the soft PVC toys (53%) were acquired at a price lower than 400 DA.

Table 5. Distribution of soft PVC toys by price (n=149).

Purchase price	Number of Items	Percentage
<200DA	22	14.8%
200-400DA	57	38/2%
400-600DA	38	25.5%
600-800DA	13	8.7%
>800DA	19	12.8%
Total	**149**	**100.0%**

II.2.5. Comparison between polymer identification techniques (flame test and IR spectroscopy)

We compared the results obtained by the two polymer identification techniques for PVC (flame test and IR spectroscopy) (Table 6).

Table 6. Comparison of results obtained by flame test and IR spectroscopy.

		Flame test		**Total**
		Positive	**Negative**	
IR	**Positive**	149	0	149
	Negative	3	119	122
Total		**152**	**119**	**271**

II.2.5. Comparison between Polymer Identification Techniques (Flame Test and IR Spectroscopy)

We identified three cases of false positives (positive results from the flame test and negative results from IR spectroscopy). The number of true negatives was 122 (negative results from IR spectroscopy). The specificity of the flame test was calculated using the following equation: **Specificity = TN / (TN + FP) = 122 / (122 + 3) = 0.976.** The specificity of the flame test was 97.6%.

DISCUSSIONS

III.1. Identification of the chemical nature of the polymer

The chemical nature of the polymer in the toys was identified through the flame test and confirmed by IR spectroscopy. Each toy was subjected to a flame test, a non-specific assay aimed at detecting the presence of chloride ions in the products. The detection of these ions allows for the elimination of toys with compositions differing from that of PVC [4, 5].

Out of the 271 toys we selected, 152, equivalent to 56.1%, yielded a positive result in the flame test, while 119 toys, or 43.9%, returned a negative result. IR analysis was employed as a confirmation method to support the positive results obtained from the flame test. Following the IR analysis, it was established that out of the 271 examined plastic toys, 149 were made of PVC-type polymer, while 122 were made from materials other than PVC. The relative frequency of soft PVC toys was 55%. PVC is one of the most commonly used polymers in the manufacture of plastic toys [3].

A study conducted by Njati SY et *al*. (2019) demonstrated that PVC materials predominate in the production of soft plastic toys intended for children [14]. The relative frequency of PVC toys was estimated at 55% in our study. Several studies have revealed similar results. Al-Natsheha M et *al*. (2015) found that 63% of the 73 toys collected from the Jordanian market were made of PVC, with the chemical nature of the polymer determined by the flame test [6]. Similarly, in an assessment conducted by ANSES in 2016, the chemical nature of the polymer was identified through the flame test, with results confirmed by IR-ATR. Among the 21 toys examined, 16 were made of PVC, representing a relative frequency of 76.2% [15].

Other studies have reported a lower frequency of PVC toys. The study conducted by Babich MA et *al*. (2020) tested 63 plastic toys, comprising a total of 129 pieces sold in the United States. Of this total, 38 pieces were found to be made of PVC, representing a relative frequency of 30%. The chemical nature of the polymer was identified using IR-FTIR [16].

The versatility of PVC, its advantageous performance, and especially its low cost make it an ideal material for various applications [17]. Its affordability is particularly exploited in the production of plastic toys for children, as highlighted by Ruth Stringer et *al*., who assert that it is widely used in the manufacture of toys and other products intended for children [18]. Indeed, the majority of plastic toys for children are made from PVC [19].

We observed that nearly a quarter (21.5%) of the PVC toys, or 32 toys, had an unknown origin, while 117 toys (78.5%) were of Chinese origin. China remains the world's leading supplier of toys, attracting many manufacturers due to the affordable labor available in the country [20]. According to data from the Chinese Toy and Baby Products Association (TJPA), the toy industry generated exports valued at $248 billion [18].

More than half of the PVC toys come from China. This finding aligns with the results of the study conducted by Stringer R et *al*., where most of the analyzed PVC toys were also of Chinese origin [21]. Among the 271 initially selected toys, 59.1% of those from China were made of PVC, while 40.9% were not. For toys without a mention of the country of origin, 50% were made of PVC, and none of the locally produced toys were made of PVC. A statistically significant correlation was observed between the nature of the polymer in the toys and their country of origin ($p < 0.05$). This finding is consistent with the results of the study conducted by Rangaswamy J et *al*. (2018), where 70% of the toys sold in the Indian market were of Chinese origin, most of which were made of PVC [20]. Toys from China sold in

unregulated markets, without composition controls, exhibit a higher concentration of harmful chemicals. The same study revealed that PVC toys imported from China were more toxic than traditional toys manufactured in India [20].
More than half of the PVC toys (53%) were sold at a price below 400 DA. PVC toys, due to their low production cost, are generally offered at more affordable prices compared to toys made from other materials [22].
Various studies have established a link between the price of toys and their composition in toxic substances. A study conducted by Turner A et *al*. (2018) highlighted that second-hand toys from thrift stores and street vendors contained higher levels of toxic substances, and these substances were present at higher frequencies than in new plastic toys purchased from department stores [23].

II.2. Comparison of two techniques for identifying pvc polymers
The table 7 provides a comparison between the two methods used for identifying the chemical composition of the polymer.

Table 7. Comparison between the Flame Test and IR Spectroscopy [10, 24].

Technique	Flame Test	IR Spectroscopy
Advantages	- Easy to handle. - Low cost. - Highly sensitive. - Eliminates non-PVC samples, reducing the size of the sample for IR analysis.	- Specific. - Very sensitive. - Determines the composition of PVC additives (plasticizers, pigments, *etc*.). - Data processing is straightforward. - Versatile, quick, and reliable technique. - Applicable in the analysis of aqueous solutions or solid samples. - High linearity range. - High frequency precision. Mechanical simplicity. - High signal-to-noise ratio.
Limitations	Lacks specificity.	- Expensive equipment. - Requires pre-treatment. - Useful for organic compounds. - Lacks a diverse library of spectra.

In our study, we obtained 122 true negatives and only three false positives. The specificity of the flame test was estimated at 97.6%, meaning that the probability of obtaining a negative result when the toy is not made of PVC using the flame test is 97.6%. This specificity is relatively high [13]. These results are supported by a study from the Canadian Conservation Institute, highlighting that the *Beilstein* test is specific to chlorinated products [5]. Among the common substances that produce positive results with the flame test, it is important to mention PVC, polyvinylidene chloride, chlorinated rubbers, chlorinated epoxy resins, and chlorinated solvents, as well as any compounds containing these substances [5]. It should be noted that the *Beilstein* test can generate false positives, but it is not prone to producing false negatives [7].

Conclusion
This study allowed us to determine the relative frequency of PVC toys intended for children under 36 months in various regions of northern Algeria, revealing a significant presence of this material in marketed

products. The analysis showed that PVC remains a predominant choice in toy manufacturing, raising concerns about potential health risks for children, particularly due to the presence of certain toxic plasticizers often associated with this polymer.

The comparison between the two identification methods—the flame test and infrared spectroscopy (IR)—revealed that while IR spectroscopy is more specific and reliable for detecting PVC, the flame test offers notable advantages in terms of speed and low cost. The latter serves as a simple and efficient method for initial screening, particularly in contexts that require quick and economical assessments.

Given the high prevalence of PVC toys on the market and the potential health implications for children, it is crucial to strengthen the monitoring of these products and promote the use of safer materials in their production. Further studies on the chemical composition of plastic toys and their long-term health impacts on children are also necessary to guide toy regulation and safety policies.

Conflict of interests

There are no conflicts to declare.

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