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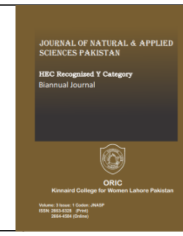
Journal of Natural and Applied Sciences Pakistan

Journal homepage: http://jnasp.kinnaird.edu.pk/



EXAMINATION OF LEAD AND CADMIUM CONTENTS IN COMMERCIAL PAINTS AND VARNISHES MARKETED IN PUNJAB PROVINCE OF PAKISTAN

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Article Info

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Keywords
Lead, Cadmium, Paints, Varnishes, Atomic Absorption Spectrometer, USEPA

Abstract

Lead and cadmium are being used in paints since ancient times. Both these metals are poisonous and cause various health and environmental irregularities. The present work was conducted to examine the lead and cadmium concentration in decorative commercial paints and varnishes marketed in Punjab province of Pakistan. Samples of fifty four paints and sixteen varnishes from different brands and three different cities were collected. Nitric and perchloric acids (3:1) of analytical grade were used for their digestion by wet acid digestion method. Atomic Absorption Spectrometer (AAS) was used to estimate the concentration of Pb and Cd which varied from 1.05 ppm to 25717.95 ppm for Pb and from 0.11 ppm to 5317 ppm for Cd in paints and from 0.06 ppm to 1.14 ppm in varnishes. The safe limits, as recommended by United States Environmental Protection Agency (USEPA) and World Health Organization (WHO), are 600 ppm (for lead) and 90 ppm (for cadmium). Only twelve paints samples were found to be safe. But in contrast to paints, all varnish samples have been found safe except two. The results may be useful for general public as well as official bodies.

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1. Introduction

Paints are usually stable emulsions of one or more different pigments, binders, some additives and solvents. The use of paints is for decoration, protection, preservation or adding functionality to any surface or target by wrapping it with a pigment coating(Roy, 1993). Pigments found naturally consist of various clays, silicas, talcs ($2MgO.4SiO_2.H_2O$) and mica ($K_2O.3Al_2O_3.6SiO_2$). Synthetically prepared pigments include polymerized silicas, calcined clays, prepared pigment molecules, blanc fixe, precipitated calcium carbonate etc. Further hiding pigments comprise red iron oxide (Fe_2O_3) titanium dioxideand phthalocyanine blue ($C_{32}H_{16}CuN_8$). Titanium dioxide (white powder, TiO_2) is a favored pigment due to its high index refraction and greater hiding power (Farmand, 2012). Toxic heavy metals and their compounds play a variety of roles in paints like corrosion inhibition, fire retardants, drying agents, preservatives etc. Chrome yellow (lead chromate) and white lead (lead carbonate) have excellent hiding power and widely used as high quality paint pigments. For bright colours, pigments such as blue lead and red lead are also used where colour on metals is desired alongside corrosion protection (Calafat, Ye, Silva, Kuklenyik, & Needham, 2006). In the third international labour conference of the League of Nations, white lead was banned for interior use (Rainhorn, 2013). The use of lead, especially white and others as well, has been consequently declined in paints appreciably. Regulatory changes have also been proposed both for interior and exterior consumer paints by U.S. Environment Protection Agency Standards to decrease lead in paints to 0.06% by dry weight (600 ppm)(David Binstock *et al.,* 2012) and 0.009% (90 ppm) by the consumer product safety improvement act (Public Law 110-314). Among the cadmium salts, cadmium sulphide is frequently used as yellow pigment. For red shades, cadmium selenide is used, commonly known as cadmium red. In the European Union, cadmium concentration in consumer paints is regulated/ restricted to 100 $\mu g\ g^{-1}$ (Turner, 2019).

Lead, being environmental pollutant, has long half-life and its accumulation in body may cause toxicities in many organs and tissues. Prolonged exposure of lead affects the central nervous system directly and causes serious learning retardation and worse changes in children behaviors. It also increases blood pressure and cardiovascular diseases in adults as well as responsible for kidney damages (Castaño *et al.*, 2012; Choi, Hu, Mukherjee, Miller, & Park, 2012; *Measuring lead exposure in infants, children, and other sensitive populations*, 1993).Cadmium is deposited in the human body, especially in kidneys. Accumulation of cadmium in the renal cortex of kidney prompts its crack with debilitated reabsorption of, for example, proteins, glucose, and amino acids. Its other complications include osteoporosis, prostate cancer, anosmia (*Cadmium: environmental aspects*, 1992; Choi *et al.,* 2012).

With the passage of time, the painted surfaces slowly deteriorate as the binders and pigments are degraded. Resultantly the paints begin to crack, flakes and chalk are mobilized into the environment. Children have the habit of eating chips/ dried films of domestic paints because they remain in regular contact with the painted surfaces while doing their playing activities through swinging, crawling, gripping and climbing etc. In and around our homes and other buildings, paints contribute to dust and soil contamination which is the major source of exposure of these toxic metals for human beings (Adebamowo *et al.,* 2007). Scrapping and remodeling activities of buildings are also another source of exposure towards these toxic paints. Keeping in view the toxic effects of heavy metals towards human health, numerous studies are available in literature describing the contents of these metals in paints and allied formulations. Heavy metals including Cd, Pd and Cr were analyzed from indoor dust and paint chips from residential houses in Nairobi, Kenya and the concentration of these metals came out to be 73, 290 and 78 μg/g of the paint samples respectively (Ogilo *et al.*, 2017). In another study conducted in Sudan, it was found that the paints were containing Cd, Pd & Cr in a range of 0.8-0.1 ppm for Cd, 3430-0.1 ppm for Pd and 500-0.1 ppm for Cr (Elkhidir, 2011). A recent study carried out at Ethiopia revealed that oil-based house paints intended for residential use were containing Pd > 90 ppm with highest content of 51,200 ppm (dry weight) in the orange paint samples. The levels of Cr and Ni were in the range 43-50 and 60-128 ppm (dry weight) while Cd was below detection limit in all analyzed paint samples (Megertu & Bayissa, 2020). In a global study (Kumar, 2009), amount of lead was analyzed in decorative paints collected from ten countries namely Sri Lanka, Philippines, Thailand, Tanzania, South Africa, Nigeria, Senegal, Belarus, Mexico and Brazil. A total of 317 paint samples were analysed and it was found that 53% samples were containing more than 90 ppm of Pd while 50% samples had lead concentration of more than 600 ppm. In this study it was also found that multinational paint brands used in more than one country showed variation in lead concentrations for samples sourced from different countries. A study conducted at Benghazi, Libya, showed that oil-based paints were containing lead between 292-998 ppm of lead, 3-38 ppm of cadmium and 0-61 ppm of chromium (El-Shawish *et al.,* 2015). Heavy metal contents were analysed in face and finger paints sold in Turkish markets and detection limits were found to be 7.71 μg/L for Pb, 0.21 μg/L for Cd, 0.47 μg/L for Mn and 3.52 μg/L for Ni (Erbas, Karatepe, & Soylak, 2017). Analysis of toxic metals in playground paints from South West England showed that Pb was ranging from 10 to 152,000 μg/g (dry weight) in 102 out of 242 cases whereas Cd was up to a concentration of 771 μg/g (Turner, Kearl, & Solman, 2016).In Portugal, a study of was carried out for heavy metal contents in children's play paints like "artist paints" and "face paints" commonly used in preschool establishments and commonly available at stores. The determinations were carried by atomic absorption spectroscopy and the levels obtained [mean ± SD (maximum)] were: 0.48±0.44 (1.98) μg/g for Pd; 0.04 ± 0.04

(0.30) µg/g for Cd; 0.17 ± 0.20 (1.47) µg/g for Co; 1.36 ± 2.18 (9.40) µg/g for Cr; 0.63 ± 0.56 (3.10) µg/g for Ni; 19.8 ± 88.2 (718) µg/g for Mn (Rebelo, Pinto, Silva, & Almeida, 2015).

The present study was conducted to examine the concentration of cadmium and lead in paints and varnishes marketed in Punjab province of Pakistan by employing atomic absorption spectroscopic technique.

2. Experimental

2.1 Sampling and Study Area

Samples of paint and varnish were collected from local markets of three different cities of Punjab, Pakistan: Bahawalnagar, Sialkot and Lahore (Figure 1). These are the major cities having wholesale markets from where paint is distributed to all nearby localities. The experimental work was performed in the laboratory of Geological Survey of Pakistan, Lahore and Chemistry Department, University of Education, Lahore, Pakistan.

2.2 Chemicals

Lead nitrate and cadmium nitrate (Merck) were used for the preparation of standards of lead and cadmium. Nitric acid and perchloric acid (Merck) in three to one ratio were used for the digestion of samples.

2.3 Instruments

Weighing balance (Sartorius TR-2145) was used to weigh the samples which were dried in oven (Heraeus MC-03820508). Metals were estimated by Atomic Absorption Spectrophotometer (Thermo Fisher ICE-3000).

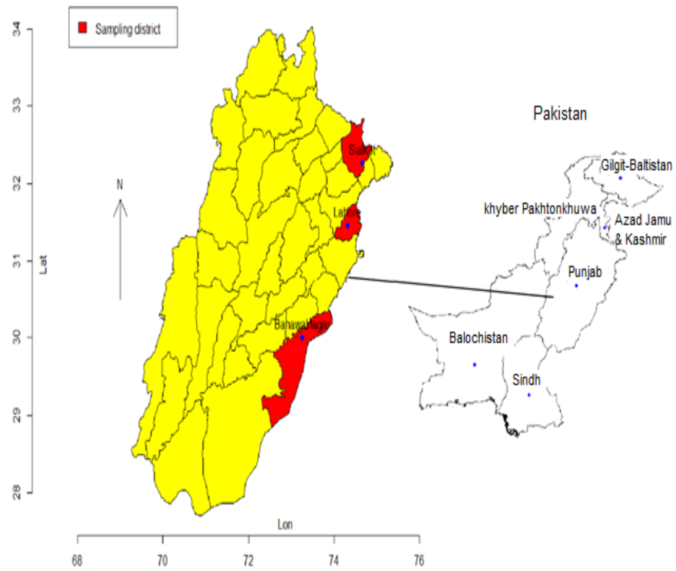

Figure 1: Map Showing the Districts for Sampling

2.4 Drying of Samples

Paint and varnish samples were shaken thoroughly to get homogeneous mixtures and poured to individual clean china dish. Samples were dried in two steps. Firstly, these were left to dry in dust free open air for at least 72 hours. In the 2nd step, these were then dried in oven for one day (24 hours) at 60 ºC. Samples were scraped off after drying by clean, sharp scalp and weighed to 1.000 g accurately using analytical balance and put into a beaker (acid washed)(DA Binstock, *et al.*, 2004).

2.5 Acid Digestion

Wet acid digestion method was adopted for the samples of paint and varnish. These were treated with perchloric acid ($HClO_4$) and concentrated nitric acid (analytical grade) to destroy the organic matter. Concentrated nitric acid (30 mL) was added to 1 g sample and digested until the formation of brown fumes was stopped at approximate temperature of 150ºC. Then after cooling, perchloric acid (10 mL) was added for absolute digestion, till the white vapors were exhausted. These digestates were then cooled to room temperature and diluted by double distilled deionized water (15 mL), filtered and further diluted to 100 mL in a volumetric flask by double distilled de-ionized water and used for further analysis (Chakrabarti, 2016).

2.6 Preparation of Standard Solutions

Standard stock solutions of cadmium and lead were prepared with a concentration of 1000 ppm. Each standard solution was diluted to 100 ppm using deionized water. The working standard solutions of 5, 10, 15 and 20 ppm of lead were made from 100 ppm diluted standard stock solution while working standard solutions of 1, 1.5 and 2 ppm of cadmium were prepared in the same way.

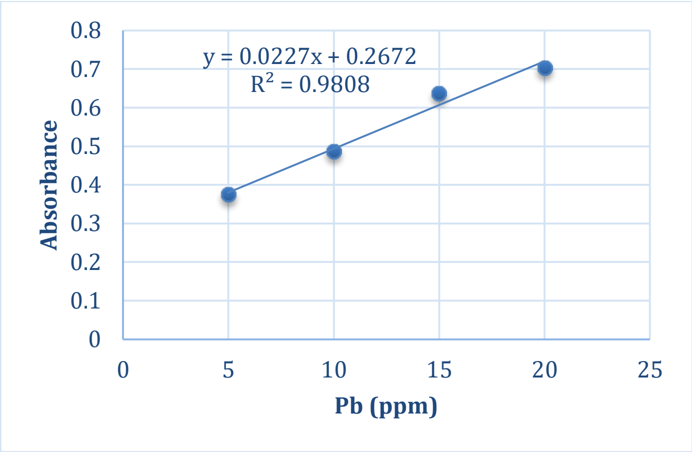

Figure 2: Calibration Plot of Lead

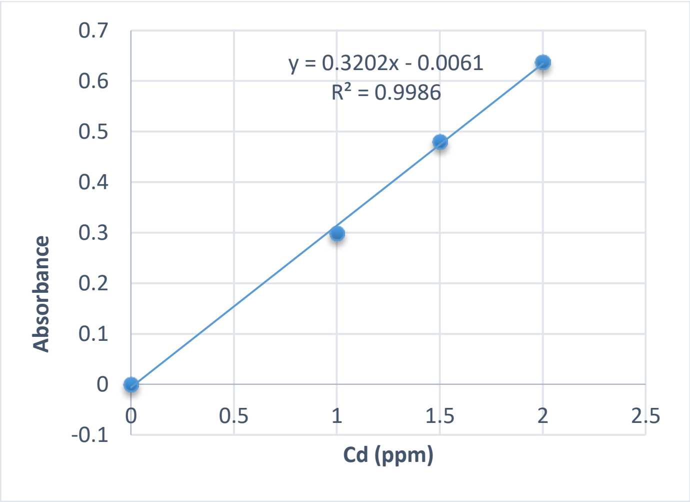

Figure 3: Calibration Curve for Cadmium

2.7 Calibration Curve and Regression Analysis

Calibration curves as shown in Figures 2 and Figure 3 were prepared from working standard solutions of lead and cadmium from their absorbance in atomic absorption spectrometer. The regression parameters for these curves are shown in Table 1.

Table 1: Regression Parameters for Lead and Cadmium with Concentrations

Elements	Regression	R_2	conc. (ppm)
Pb	Y = 0.0227X + 0.2672	0.980 8	1.0–20.0
Cd	Y = 0.3202X - 0.0061	0.998 6	1.0–3.0

2.8 AAS Analysis

The concentration of lead and cadmium was measured by Atomic Absorption Spectrometer. Validity of the method was deduced by replication analysis and internal calibration method. Working standard solutions were used for calibration of instrument. The blank samples (deionized water) and standards were repeated after running of every 20 samples. The main instrumental parameters such as lamp current,

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wavelength, measurement time, heat of the flame, band width, slit width and the sensitivity for AAS instrument were optimized separately for each metal as shown in Table 2

Table 2: Operational Parameters of AAS

Parameter (unit)	Cadmium	Lead
Wavelength (nm)	228.8	217
Measurement time (sec)	1	1
Lamp current (mA)	10	10
Slit width (nm)	0.5	1.0
Sensitivity (μg/g)	0.0110	0.1100
Flow rate (litres/mm)	1.5	1.5
Limit of detection (μg/g)	0.0006	0.0200

3. Results and Discussion

3.1 Measurement of Lead and Cadmium

Paint samples (54 in number) were analyzed in triplicate for Pb and Cd. The mean concentration of these metals in eight paint brands is given in Table 3.

Table 3: Amount of Lead and Cadmium in Brands A to H

Color	Pb (ppm)	Cd (ppm)	Color	Pb (ppm)	Cd (ppm)
	Mean ± SD	Mean ± SD		Mean ± SD	Mean ± SD
Brand A (Alpha)			Brand B (Rose)		
White	7396.45 ± 1.34	6.21 ± 0.09	White	1333.62 ± 1.51	1.74 ± 0.11
Black	8506.03 ± 1.46	BDL*	Black	113.46 ± 0.08	0.44 ± 0.02
Green	6222.22 ± 0.88	3881.80 ± 0.66	Green	3208.36 ± 2.37	0.42 ± 0.00
Yellow	25717.95 ± 1.87	BDL*	Maroon	1019.49 ± 1.28	7.42 ± 0.03
Orange	8458.13 ± 1.39	5317.07 ± 0.72	Orange	1185.11 ± 1.66	3.19 ± 0.02
Red	3380.46 ± 0.69	1.99 ± 0.07	Red	6847.48 ± 3.11	1.61 ± 0.09
Blue	5305.56 ± 0.82	BDL*	Blue	1783.15 ± 1.71	1.65 ± 0.01
Brand C (Pearl)			Brand D (Jasmine)		
White	1763.60 ± 1.79	1.45 ± 0.00	White	1.05 ± 0.00	1.00 ± 0.00
Blue	550.77 ± 0.14	10.87 ± 0.02	Black	448.57 ±0.90	3.86 ±0.02
Purple	208.34 ± 0.10	4.86 ± 0.01	Green	246.14 ± 0.07	0.60 ± 0.00
Sky Blue	169.22 ± 0.11	4.99 ± 0.03	Yellow	26.20 ± 0.03	1.56 ± 0.01
Maroon	BDL*	BDL*	Orange	278.92 ±0.77	4.01 ± 0.02
Red	4026.06 ± 2.33	1.57 ± 0.00	Red	148.68 ±0.06	0.55 ± 0.00
Brand E (Amber)			Brand F (Theta)		
White	1389.42 ± 0.47	1270.81 ± 0.39	White	16.91 ±0.02	BDL
Black	926.14 ± 0.62	924.58 ± 0.51	Silver	69.21 ±0.03	2.39 ±0.00
Green	617.99 ± 0.33	25.38 ± 0.02	Grey	1968.47 ±1.66	BDL
Yellow	22.53 ± 0.03	0.48 ±0.00	Yellow	639.15 ± 0.88	0.56 ±0.00
Orange	212.16 ±0.33	0.88 ±0.02	Orange	940.50 ±1.10	883.56 ± 0.91
Blue	139.80 ± 0.21	2.71 ± 0.01	Blue	4.59 ±0.01	24.66 ±0.02
Brand G (Rho)			Brand H (Star)		
White	815.19 ± 0.17	9.51 ±0.04	White	916.27 ± 0.67	747.73 ± 0.43
Green	175.22 ± 0.04	180.92 ±0.06	Green	2993.91 ± 1.21	0.64 ±0.00
Orange	13.60 ± 0.01	BDL*	Orange	381.24 ± 0.41	1.42 ±0.01
Yellow	98.47 ± 0.05	0.13 ± 0.00	Yellow	1011.02 ± 0.73	885.57 ± 0.48
Silver	1542 ± 0.55	BDL*	Blue	666.77 ± 0.45	0.11 ± 0.00
Red	194.47 ±0.04	BDL*	Silver	53.18 ± 0.03	2.81 ± 0.01
Brown	BDL	0.90 ±0.01	Yellow Green	2025.97 ±0.99	BDL*

*Below Detection Limit

Brand A, B & C were collected from Lahore; Brand D, E, & F from Sialkot; Brand G & H from Bahawalnagar

The results of eight brands A to H indicate that the highest concentration of lead was found in yellow (Brand A), red (Brand B), red (Brand C), black (Brand D), white (Brand E), grey (Brand F), silver (Brand G) and green (Brand H) paint in contrast to the lowest detectable concentration was found in red (Brand A), black (Brand B), sky blue (Brand C), white (Brand D), yellow (Brand E), blue (Brand F), orange (Brand G) and silver (Brand H) respectively. The recommended limits of lead concentration in paint should be 600 ppm (Ettinger *et al.,* 2002).

On the other hand, the results of brands A to H indicate that the highest concentration of cadmium was found in orange (Brand A), maroon (Brand B), blue (Brand C), orange (Brand D),white,(Brand E), orange (Brand F), green (Brand G) and yellow (Brand H) paint while the lowest detectable concentration was found in red (Brand A), green (Brand B), white (Brand C), red (Brand D), yellow (Brand E), yellow (Brand F), yellow (Brand G) and silver (Brand H) respectively. The recommended limits of cadmium concentration in paint should be 90 ppm (Widman, 2010).

3.2 Statistical Analysis

The amount of lead and cadmium from both paints and varnishes are tabulated as mean concentration profile together with their standard deviation. Mean concentrations for the brands are further analyzed for their standard error. To compare differences in areas of Punjab with respect to lead and cadmium in paints and varnishes are also determined by analysis of variance (ANOVA).The significant level was set at $p = 0.05$ ($\alpha = 0.05$). The ANOVA is useful in providing interdependence of variables and significant differences in the paint brands as representative samples(Churchill, 2004)

The concentrations of lead were significantly different from each other among different brands of Lahore. However, results for Sialkot and Bahawalnagar were found non-significant. Statistically, brands of all three districts were not different from each other for cadmium concentrations.

3.3 Statistical Analysis of Paint Samples

The said statistical analysis for various brands of paints from different cities of Punjab province is shown in Table 4

Table 4: Analysis Of Variance of Concentrations of Lead (Ppm) And Cadmium (Ppm)

Source of variations	df	MS (Pb)	MS (Cd)
Brands from Lahore	2	156503.29*	34879.72ns
Brands from Sialkot	2	1445.08ns	1231.89ns
Brands from Bahawalnagar	1	345.98ns	21717.25ns

df=degree of freedom, MS=Mean Square, ns=non-significant

3.4 Analysis of Lead and Cadmium in Varnishes

Varnish samples (16 in number) were analyzedin triplicates for cadmium and lead as shown in Table 5. These samples were collected from four different cities of Punjab province of Pakistan.

Table 5: Mean Values of Cd and Pb Concentrations Of Different Brands Of Varnishes

Location	Brand	Cd (ppm)	Pb (ppm)
Sialkot	ML	0.33 ±0.02	22.33 ±0.08
	BO	BDL	b8.81 ±0.01
	CE	BDL	1859.64 ±1.54
	DW	BDL	361.24 ± 0.49
Lahore	TM	1.14 ±0.02	6.72 ± 0.07
	GF	0.20 ±0.00	1.10 ± 0.00
	PL	0.23 ±0.01	3.98 ± 0.01
	HC	0.05 ±0.00	4.85 ±0.01
Bahawalnagar	HS	0.87 ±0.4	1.66 ± 0.03
	DD	0.16 ±0.01	0.69 ±0.01
	SE	0.09 ±0.00	0.80 ±0.02
	GS	0.12 ±0.00	1.17 ±0.02
Bahawalnagar	PX	0.06 ± 0.00	0.60 ± 0.01
	BB	0.04 ± 0.01	2.21 ± 0.02
	SL	0.28 ± 0.02	2.05 ± 0.00
	NC	0.87 ± 0.01	0.22 ± 0.01

Means with the same letters in each column and each row do not differ significantly at the 5% level.

CE varnish sample found to have the highest lead concentration (1859.64 ppm), on the other hand, NC varnish sample had lowest lead concentration (0.22 ppm).

Varnish sample of TM brand had the highest cadmium concentration (1.14 ppm) while BB varnish sample found to have lowest cadmium concentration (0.04 ppm).

Lead and cadmium concentration in almost all varnish samples was found to be within WHO recommended safe limit (90 ppm).

4. Conclusion

The results show that many of the commercially available local paints used in Pakistan found to have higher concentration of lead and cadmium above the WHO recommended safe levels (600 ppm for lead and 90 ppm for cadmium) in almost all the paints analyzed. It is noted that concentration of lead is much higher than cadmium in paints and it is different in various analyzed brands. Significance dissimilarity is found in the concentration of lead and cadmium in analyzed paints having color of red, orange, black and white whereas these metals were found within WHO recommended safe limit in almost all varnish samples.

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