

Quantitative Analysis of Heavy Metals in Used Lubricant Oil (ULO) Collected from Quetta City, Pakistan

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Abstract

The disposal of Used Lubricating Oil (ULO) is an emerging global environmental issue, especially in developing nations where waste management systems and regulatory frameworks are weak. ULO is a complex mixture of toxic heavy metals coming from wear and additive breakdown, which can have detrimental effects on soil and water quality. In this research, the levels and sources of heavy metals in ULO from four typical vehicles in Quetta City, Pakistan, were studied using energy-dispersive X-ray Fluorescence (XRF) spectroscopy. Nine elements (Pb, Sn, Cu, Ni, Cr, Mn, Zn, P, and S) were measured, with high levels of Tin (50,000-117,800 mg/kg) and Copper (11,100-31,600 mg/kg) suggesting significant wear. Zinc (777-867 mg/kg) and phosphorus (908-1,046 mg/kg) exhibited minimal variations, indicating uniform additive compositions. Principal component analysis (PCA) revealed two major sources of variability (91.2% variance explained), including wear contaminants (bearings, bushings, and unburnt fuel) and additives, Zinc Dialkyldithiophosphate (ZDDP-based additives). Compared with international soil quality guidelines, the levels of Tin (Sn), Copper (Cu), Nickel, and Chromium (Cr) are well above the recommended limits, suggesting environmental concerns associated with improper ULO disposal. While Lead concentrations were found to be low due to the success of lead-free fuel policies, the study highlights the importance of collecting ULO, enforcing regulations, and undertaking environmental surveillance. This research provides a baseline assessment of ULO contamination in Pakistan and contributes to international knowledge of ULO contamination, thereby aiding the establishment of sustainable waste-oil management practices.

Keywords: XRF; Used lubricating oil, Heavy metals; Source apportionment; Environmental risk; Quetta

1. Introduction

Used lubricating oil (ULO) is a major environmental issue. Lubricating oil deteriorates during engine operation and contains hydrocarbons, wear metals, and residual additives [1]. Many of these compounds are resistant to degradation, and some are carcinogenic and toxic [2]. Inappropriate disposal of ULO, such as spilling on soil, into drainage, or with solid waste, can result in environmental pollution. In less developed regions like Pakistan, enforcement of regulations is weak, and disposal infrastructure is limited [3]. The major sources of heavy metals in ULO include: First, engine wear emits metals from components such as bearings (lead, Tin), bushings (Copper, Tin), piston rings (chromium), and steel alloys (nickel). Second, ULO additives (mainly zinc dialkyldithiophosphate, ZDDP) add other elements such as zinc and phosphorus with anti-wear and antioxidant properties [4,5]. Metals analysis can offer insights into engine health and potential environmental hazards [6]. Several techniques are used worldwide to measure heavy metal levels in ULO, including atomic absorption spectrometry (AAS), inductively coupled plasma optical emission spectrometry (ICP-OES), and X-ray fluorescence (XRF) [7,8].

Used lubricating oil (ULO) must be managed throughout the total product lifecycle (production, use, reuse, or disposal). Thousands of tons of lubricating oil are used worldwide each year, much of which becomes used lubricating oil (ULO) that needs to be managed. Many developed nations have an established infrastructure for collection, recycling, and refining to prevent adverse environmental effects. But in many developing countries, such as Pakistan, the ULO is disposed of casually by being dumped on land, into drainage systems, or as waste. Disjointed lifecycle management of ULO results in the accumulation of toxic metals in the environment and underscores the need for holistic, sustainable waste-oil management approaches that incorporate technological, regulatory, and behavioral measures.

XRF has several advantages, including high speed, minimal sample preparation, non-destructive analysis and multi-element determination [9,10]. Official methods such as ASTM D6481 and ASTM D7751 are often used for the elemental analysis of lubricants [11-12]. This research offers an initial study of heavy metal contamination in ULO from Quetta, Pakistan. The study seeks to assist local management in making policies by assessing local oil use and disposal practices [13]. The research identifies potential risk factors and provides a basis for expanding this type of assessment to other urban areas in Pakistan, thereby guiding ULO management strategies at the national level.

2.0. Materials and Methods

2.1 Chemical and Reagents

All CRMs were purchased from commercial providers. NIST SRM 1085c was used for wear metals (Pb, Sn, Cu, Ni, Cr, Mn). VHG Labs V21-50-250 was used for additives (Zn, P, S). Polypropylene film (4 µm thick, XRF grade) was bought

from Oxford Instruments. New lubricant oil (blank sample) was purchased from Quetta. Sample cups and bottles were pre-cleaned with 10% HNO₃ solution, followed by Deionized water.

2.2. Sample Collection

Four samples of used lubricating oil (ULO) were taken from four different vehicles at four workshops in Quetta City. Approximately 500 mL of sample was drained from each vehicle in acid-washed polyethylene bottles. The samples were marked, brought to the laboratory, and mixed for analysis. Vehicle details are in Table (1).

Table 1: Vehicle information for each sample.

Sample	Vehicle	Year	Engine Type	Location	Mileage (km)
A	Suzuki Cultus	2003	Gasoline (1.0 L)	Satellite Town	145,000
B	Toyota Corolla 2D	2004	Gasoline (1.3 L)	Airport Road	168,000
C	Jeep Potohar	2007	Diesel (2.5 L)	Jinnah Town	210,000
D	Suzuki Alto VXR	2004	Gasoline (0.8 L)	Sariab Road	132,000

2.3. Sample Preparation

For each sample, 10 mL of well-mixed ULO was transferred to an XRF sample cup with a Poly-M XRF sample film (Oxford Instruments), a high-purity, uniform window material with low background interference. The sample was filled to the mark line (approximately 13 mL capacity). The autosampler had secondary safety windows to protect the instrument from sample leaks. Analysis was done under a helium purge to enhance sensitivity for light elements (sulfur and phosphorus). The instrument was equipped with a 10-position autosampler for unattended multiple analyses [11]. Sample preparation followed the methodology described by other studies [9] for XRF analysis of engine oils.

2.4. X-Ray Fluorescence (XRF)

X-ray fluorescence (XRF) analysis was carried out with an X-Supreme 8000 X-ray fluorescence spectrometer (Oxford Instruments, UK) with Focus SD technology [11]. It features high spectral resolution from a Silicon Drift Detector (SDD), excellent elemental excitation and matrix correction using an Oxford Instruments titanium-target X-ray tube, and background filters. The combination of these features provides optimal analysis speed, minimum detection limits, and outstanding performance for multi-element analysis in ULO [11]. ASTM standard test method D7751 provides the framework for the determination of additive elements in lubricating oils by EDXRF [14].

2.5. Calibration and Quality Control

Calibration was done with reference materials:

NIST SRM 1085c for wear metals (Pb, Sn, Cu, Ni, Cr, Mn)

VHG Labs V21-50-250 for additive elements (Zn, P, S)

Recovery ranged from 95-105%. The X-Supreme 8000 was set up with parameters optimized for the analysis of used lubricant oil. For sulfur analysis, the "XSP-Sulfur" application package includes three method templates: Ultra Low (3-150 mg/kg), Medium (0.015-0.5% m/m), and High (0.5-5% m/m) (Table 2) [11]. The calibration method aligns with other EDXRF methods for oil analysis, where empirical calibration using matrix-matched standards is suggested for good results [10,14].

Table 2: Guaranteed detection limits (3 σ , as per manufacturer specifications [11]):

Element	Detection Limit (mg/kg)
Pb	1
Sn	13**
Cu	2
Ni	1
Mn	1
Zn	2*
P	12*
S	8*
Cr	1

*Converted from % m/m (Zn: 0.0002%, P: 0.0012%, S: 0.0008%) using 1% m/m = 10,000 mg/kg.

**Limit of Detection (LOD) for Sn with no spectral interference [11].

The quality control procedures included:

Setting up Samples (SUSs): Pure light mineral oil (user-supplied) and SU-S20B or SU-S40D disks with known sulfur quantities, measured during calibration as reference samples for long-term sensitivity checks [11].

Quality Control (QC) sample: CONOSTAN S21-300 measured every 10 samples; system recalibrated if it was outside control limits [11]. The use of certified reference materials for QC in XRF analysis of oils has been confirmed by other studies [9,10].

Smart Check software: Used to confirm the conformity of the analytical measurements, including large discrepancies between duplicate oil samples and that the samples were within the calibration range [11].

Blank correction with new lubricant oil.

Daily recalibration using SUSs when QC was outside tolerances [11].

2.6 Principal Component Analysis (PCA)

Principal component analysis (PCA) was used to determine the sources of heavy metals. Multivariate statistical analysis based on PCA is a data reduction technique that transforms a set of correlated variables into a smaller set of uncorrelated variables (principal components) that account for most of the variance [13]. PCA is based on the eigen decomposition of the covariance matrix or the Singular Value Decomposition (SVD) of the data matrix. If two elements come from the same source (e.g., bearing wear), they are correlated across samples and load highly onto the same component. PCA has been successfully applied to oil spectrometric data. Previous studies [12] performed PCA on 69 oil samples from a diesel engine, showing that PCA naturally groups the elements into three distinct clusters: wear metals (Fe, Cr, Cu, Pb), high-concentration additives (Na, Zn, P, Ca), and base oil elements. They demonstrated that PCA can provide insight into the sources of various elements and assess engine wear [12]. Likewise, previous studies [13] offer an overview of PCA and its use in chemometrics.

In the present paper, the following steps were performed: First, the data matrix (4 samples $\times$ 9 elements) was z-transformed (mean = 0, standard deviation = 1) to remove the influence of units (concentrations were reported in different magnitude orders: mg/kg for metals and wt. % for some additives). Second, the covariance matrix of the standardized data matrix was calculated. Third, we retained only components with eigenvalues >1 using the Kaiser criterion [13]. Loadings higher than 0.7 were used to interpret the sources [12].

3.0. Results and Discussion

3.1. Additive elements (zinc and phosphorus) showed little Coefficient of Variation (CV): Zn: 777–867 mg/kg (CV = 4.7%); P: 908–1,046 mg/kg (CV = 5.7%) as shown in Figure No. 1. This indicates that the vehicles have similar additives, consistent with previous lubricant studies, and that the base oils supplied to the vehicle owners are likely from the same company. The low coefficient of variation (CV $< 6\%$) indicates that ZDDP additives are consumed at similar rates for different engine designs and operating conditions [5].

3.2. Wear metals exhibited high inter-vehicle variability: Sn: 50,000–117,800 mg/kg (CV = 32.6%); Cu: 11,100–31,600 mg/kg (CV = 39.5%); Cr: <500 –13,500 mg/kg (CV = 97.6%); Mn: 6,300–14,100 mg/kg (CV = 35.0%); Ni: 800–5,500 mg/kg (CV = 70.3%); Pb: 100–600 mg/kg (CV = 72.0%) as shown in Figure. No 2. These large CV values, especially for Cr (97.6%) and Pb (72.0%), suggest considerable variability in engine wear, which is dependent on vehicle age, driving habits, and maintenance practices, rather than on mileage, as reported in tribological studies [1,6].

The highest Sn (117,800 mg/kg) and Cu (31,600 mg/kg) were detected in sample A (2003 Suzuki Cultus, Satellite Town), suggesting severe wear of both bearing and bushings. This car is the oldest (18 years old) and was driven on Satellite Town's unpaved roads, known for heavy traffic. This probably contributed to the faster wear of copper-based bushings and tin-based bearing overlays. This result is in accordance with studies [1] that showed that bearing wear metals increase exponentially after 150,000 km.

The highest concentration of Cr was found in sample D (2004 Suzuki Alto, Sariab Road) (13,500 mg/kg), suggesting wear of the piston ring or cylinder liner. Hard-coated piston rings and liners often contain Cr. The high Cr in this sample indicates that the engine experienced poor lubrication and/or increased particulate contamination, likely due to lack of air filter cleaning in the dusty Sariab Road area.

Sample C (2007 Diesel Jeep, Jinnah Town) had the lowest wear metals, despite the highest mileage (210,000 km). This observation can be explained by the shortest oil change interval (4 months). Regular oil changes prevent the build-up of wear particles to toxic levels. This finding is significant: frequent oil changes (every 3–4 months or 5,000 km) can greatly reduce engine wear and environmental impacts from the waste oil.

Sulfur (10,338 mg/kg) was elevated, as is normal for diesel engines, as shown in Figure (1), due to historically higher sulfur levels in Pakistan's diesel fuel [3]. Lead levels were very low (100–600 mg/kg) in all samples, around 75% lower than in European studies conducted in the early 2000s (e.g., 400–2,500 mg/kg [2,7]). This is an indication of the successful removal of leaded gasoline from Pakistan in 2016 [3,8]. Before the ban, ULO in Pakistani vehicles showed lead concentrations up to 2,500 mg/kg [8]. This significant decrease shows the impact of effective policy actions.

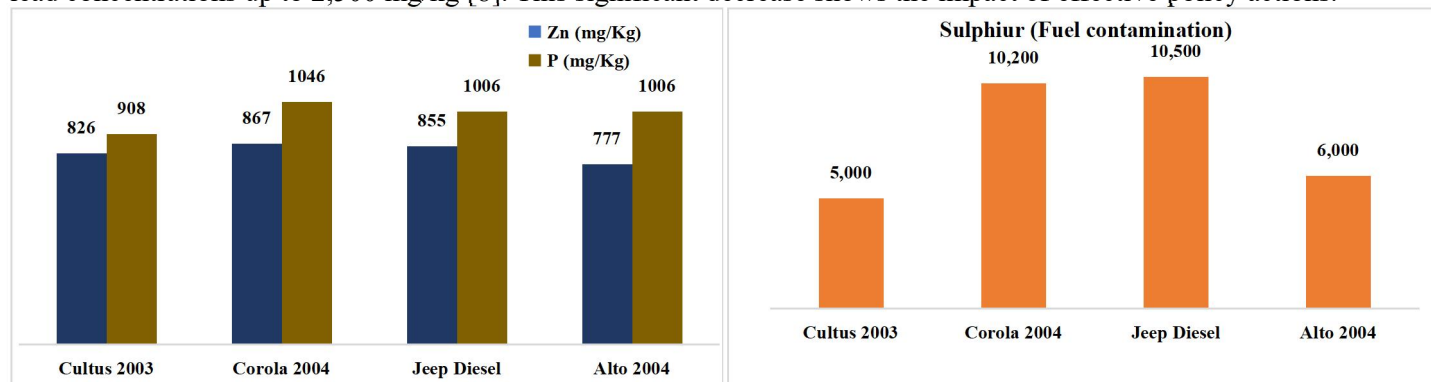


Figure 1. Distribution of Zn, P, and S concentrations showed a fuel contamination alert (10,000 mg/Kg) in ULO samples.

The concentrations of nine elements in the four ULO samples (all in mg/kg) has been added in Table (3). A comparison of results observed in the present study to other global studies, the concentrations of Tin and Copper found in this study are significantly higher than those reported in studies conducted in Europe and certain Asian countries [2,7], where concentrations of Tin in ULO are typically reported to be less than 80,000 mg/kg and those of Copper less than 20,000 mg/kg under normal operating conditions. This could be due to variations in maintenance practices, oil change frequency, fuel quality, and road conditions. On the other hand, the concentrations of zinc and phosphorus are comparable with international studies, due to the worldwide use of ZDDP additives in commercial engine oils. These findings suggest that global standardization of additive chemistry is maintained, but contamination from wear metals is highly localized and related to operating conditions and maintenance practices.

Table 3: Elemental Concentrations in ULO Samples (All Units in mg/kg)

Element	Sample A (Cultus)	Sample B (Corolla)	Sample C (Jeep Diesel)	Sample D (Alto)	Mean	SD	CV (%)
Pb	600	200	100	300	300	220	72.0
Sn	117,800	88,700	50,000	96,200	88,180	28,740	32.6
Cu	31,600	19,900	11,100	26,600	22,300	8,820	39.5
Ni	5,500	800	1,500	4,500	3,080	2,160	70.3
Cr	3,600	6,700	<500*	13,500	5,950	5,810	97.6
Mn	14,000	6,300	14,100	8,200	10,650	3,730	35.0
Zn	826	867	855	777	831	39	4.7
P	908	1,046	1,006	1,006	992	57	5.7
S	4,814	10,163	10,338	6,240	7,889	2,648	33.6

*Below detection limit (500 mg/kg)

The PCA identified two major sources of heavy metals, accounting for 91.2% of the Variance (Table 4). The fact that these two sources explain a very large percentage of the total variance suggests that they account for nearly all of the variability in the data, with the remaining 8.8% being due to measurement error or other minor, unidentified sources. All Statistical analyses were performed using R Software.

Given the small sample size (n=4), PCA results should be considered exploratory rather than definitive.

Table 4: PCA Results showing variance and loadings.

Component	Variance Explained	Elements with High Loadings (>0.7)	Interpretation
PC1	61.3%	S (0.92), Pb (0.89), Cu (0.88), Sn (0.85), Ni (0.76)	Wear and contamination source (bearings, bushings, fuel).
PC2	29.9%	Zn (0.91), P (0.65)	Additive source (ZDDP anti-wear)
Total	91.2%		

3.3 Environmental Risk Assessment

Table 5 compares the concentrations of metals in Used Lubricating Oil (ULO) with some international soil standards [14-16]. These are used only for screening environmental risk, rather than for direct comparison, due to the different matrices (liquid ULO vs. solid soil).

The main focus of environmental concern is on Tin. In three of the four samples (75%), the value is above the proposed European soil standard (50,000 mg/kg) [14]:

Sample A (Satellite Town): 117,800 mg/kg (2.4× guideline)

Sample B (Airport Road): 88,700 mg/kg (1.8× guideline)

Sample D (Sariab Road): 96,200 mg/kg (1.9× guideline)

Tin is moderately toxic to aquatic organisms, with EC50 values for algae ranging from 10 to 100 mg/L [12]. High tin content in ULO indicates that, unless disposed of carefully, ULO can be a potential source of soil and water contamination. In Quetta, used oil is often disposed of on dry land or in drains that lead to seasonal streams (karezes), posing a potential environmental risk to groundwater recharge zones and downstream water contamination.

Lead levels (100-600 mg/kg) are not as high as those reported from areas where leaded gasoline was previously used. However, compared to recently proposed residential soil screening levels (200 mg/kg), Sample A (600 mg/kg) is above this threshold and may need site-specific assessment [15]. While lead concentrations are generally low, there is potential for occupational exposure if not used in well-ventilated areas. Personal protective equipment and better ventilation are therefore advised.

Copper levels (11,100-31,600 mg/kg) are much higher than recommended soil levels (50-300 mg/kg, depending on soil characteristics), and may pose an ecological risk if released into the soil environment [14,16]. Increased copper concentrations can impact on soil organisms, such as earthworms and microorganisms [12].

Nickel levels (800-5,500 mg/kg) are also higher than typical soil guidelines (30-75 mg/kg) [14]. While some nickel compounds are carcinogenic, their carcinogenicity depends on nickel chemical speciation, mode of exposure, and bioavailability [16]. Therefore, the results indicate a potential carcinogenic risk.

Manganese (Mn) levels ranged from 6,300 to 14,100 mg/kg, with a coefficient of variation (CV) of 35.0%, indicating moderate variability among the samples. Mn is normally associated with alloy components in engine steels and, in some

instances, is also linked to fuel additives [1,6]. High levels of Mn indicate gradual corrosion of steel engine components, including shafts and gears. Environmentally, overaccumulation of manganese in soil can affect microbial activity and plant growth, and long-term exposure in humans has been reported to have neurological effects [16]. Even though Mn is a necessary trace element, its high levels in ULO support the idea that controlled disposal is the only way to prevent Mn accumulation in the environment. Mn has a relatively uniform distribution compared to other wear metals.

Chromium concentrations (as high as 13,500 mg/kg) exceed other soil guideline values (100-150 mg/kg) [14]. However, the toxicity of chromium depends on the oxidation state - hexavalent Cr (Cr (VI)) is more toxic than trivalent Cr (Cr (III)) [17]. Therefore, speciation studies are required to determine risk. These elevated total chromium concentrations indicate further research is needed, particularly in areas of concern (Satellite Town and Sariab Road).

Table 5: Environmental Risk Assessment (All Units in mg/kg)

Metal	This Study	US EPA SSL [15]	EU Guideline [14]	Risk Level
Pb	100–600	200	300	Low–Moderate
Sn	50,000–117,800	Not available	50,000*	High
Cu	11,100–31,600	3,100	50–300	High
Ni	800–5,500	1,600	30–75	Moderate–High
Mn	6300–14,100	1800	500–1000	Moderate
Cr	<500–13,500	210	100–150	Moderate–High
Zn	777–867	23,000	150–300	Low

* Proposed (non-binding) European guideline value for Tin [14]

This study focuses on identifying the pollution potential of ULO rather than its environmental impacts. The lack of soil and water sampling is a limitation of this work; however, the presence of elevated heavy metals in ULO indicates potential risk. Thus, this study is a preliminary one to inform future research, which will include integrated environmental sampling and risk assessment.

3.4 Policy Gap Analysis

This study raises concerns about the environmental consequences of improper disposal of used lubricating oil (ULO), but did not involve environmental sampling (soil and water contamination levels). Additionally, with poor regulatory enforcement in Pakistan, this study does not include an in-depth assessment of Pakistan's environmental policies and enforcement practices. There is a need to link environmental monitoring with policy analysis to improve evidence-based policy-making. In particular, Researchers should:

Assess the concentrations of Tin, Copper, and Chromium in water and soil samples from around the workshops, with a key focus on hotspot areas such as Satellite Town (Sample A) and Sariab Road (Sample D).

Speciation of chromium to determine the presence of the more toxic hexavalent chromium (Cr (VI)) and less toxic trivalent chromium (Cr (III)). Evaluation of the existing environmental laws in Balochistan in terms of institutional framework, compliance, and enforcement.

3.5 Recommendations

The risk assessment and results suggest the following measures:

Short term (0-3 months): Alert Satellite Town and Sariab Road workshops to the risk. Provide ULO collection bins. Conduct baseline soil testing around at-risk workshops.

Medium-term (3-6 months): Establish ULO collection and storage facilities in Quetta. Train mechanics to safely dispose of waste oil and to perform regular oil changes (e.g., every 4 months or 5,000 km). Educate the community about the health and environmental harms of ULO.

Long-term (6-12 months) - Conduct a feasibility study of a local (small) ULO re-refining industry in Quetta. Develop a regulatory mechanism with Extended Producer Responsibility (EPR). Analyze a larger number of soil and water samples (20-25). Perform chromium speciation in contaminated samples.

4.0 Conclusion

In the current study, the concentration and source of heavy metals in used lubricating oil (ULO) collected from Quetta were evaluated using X-ray fluorescence (XRF) spectroscopy and principal component analysis (PCA). The findings demonstrated that the wear-related metals had a high level of variability particularly among the metals such as Sn (50,000- 117,800 mg/kg), Cu (11,100-31,600 mg/kg), Cr (<500-13,500 mg/kg), Ni (800-5,500 mg/kg), and Mn (6,300-14,100 mg/kg) due to the differences in the conditions of the engine, its maintenance and the operating environment. Conversely, additive-derived elements, including Zn (777-867 mg/kg) and phosphorus (908-1046 mg/kg), had low variability, indicating that the same lubricant formulations were used across the vehicles. The PCA showed that two dominant sources explain 91.2 % of the total variance: (i) wear and contamination sources related to engine components (Pb, Cu, Sn, Ni, Cr, Mn, and S), and (ii) additive-related inputs mostly associated with zinc dialkyldithiophosphate (ZDDP). These results affirm that the process of ULO composition is controlled by both mechanical wear and lubricant chemistry. The environmental risk assessment revealed that several metals, especially Sn, Cu, Cr, and Ni, exceed the values of the international soil guideline, indicating that they have a high potential to contaminate the environment if

disposed of improperly. Manganese was also reported to have high levels, which might lead to ecological and human health hazards in the case of prolonged exposure. Even though leaded fuel levels were relatively low due to the phasing out of leaded fuels, there are still localized exceedances that should be considered. In general, this research indicates that ULO in Quetta is a focal point of heavy metal contamination. These contaminants may pose long-term environmental risks without proper collection, recycling, or disposal mechanisms. The results emphasize the importance of formulated approaches to ULO management, regulatory enforcement, and enhancing public awareness to reduce contamination and promote sustainable waste-oil handling practices.

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Author's Contribution:

Nauman Afridi: Conceptualization, investigation, methodology, formal analysis, data curation, validation, writing original draft, editing. Nizam-ud-din Baloch: Supervision, review, validation. Samar Ali: Supervision, review, validation. Muhammad Adnan Afridi: Investigation, data curation. Muhammad Faheem: Formal analysis, validation. Maria Saba: Literature search, data curation. Dua Nizam: Visualization, editing. Muhammad Qasim Mazari: Facilitation, resources.

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