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Multiresidue Analysis of Organochlorine and Organophosphate Pesticides in Bovine Milk Using Modified QuEChERS Method by GC-MS

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ABSTRACT

The present study was undertaken for the determination of the residual concentration of OCPs and OPPs in milk by GC-MS. In the present study, residues of OCPs viz. α, β, γ and δ-BHC, heptachlor, aldrin, heptachlor epoxide, endosulfan-I, cis-chlordane, trans-chlordane, p, p-DDE, Dieldrin, Endrin, Endosulfan-II, p, p-DDD, Endrin aldehyde, Endosulfan sulphate, p, p-DDT, Endrin ketone and methoxychlor were evaluated in milk samples. Dichlorvos, Mevinophos, Demeton, Ethoprophos, Naled, Phorate, Diazinone, Disulfoton, methyl parathion, Ronnel, Chlorpyrifos, Fenthion, Trichloronat, Stirifos, Tokuthion, Merphos, Fensulfothion, Bolstar, Azinophos-methyl and Coumaphos residues belonging to the organophosphate group of pesticides were also evaluated in these samples.

Sample extraction was performed by adopting the modified QuEChERS principle and cleaned up using a dispersive solid-phase extraction C-18 cartridge. The performance of the GCMS method was investigated for linearity, accuracy, precision, detection limit and quantification limit. Good linearity with correlation coefficients (r^2) ranging from 0.996 to 0.999 was obtained for various organochlorine components. The mean recoveries for all OCPs and OPPs components ranged between 72% to 108% and 71% to 101%, respectively.

From the total collected 70 milk samples, 5 samples from each target aera were tested for various OCPs and OPPs residues. In this study observed that 11.43% (4/35) of the milk samples tested positive for Endosulfan-I, Endosulfan-II, heptachlor, α-BHC and δ-BHC. The contamination percentages for p, p'-DDD, methoxychlor, endrin, heptachlor epoxide and p, p-DDE were 22.86% (8/35), 17.14% (6/35), 14.29% (5/35), 8.57% (3/35) and 5.71% (2/35) respectively. On the other hand, Mevinphos, Ethoprophos, Azinphos-methyl, Chlorpyrifos and Coumaphos were found to be present in approximately 25.71% (9/35), 28.57% (10/35), 20.00% (7/35), 31.43% (11/35) and 51.43% (18/35) of the milk samples, respectively.

Keywords: OCPs; OPP; GCMS, MRL; PPB; QuEChERS and residual concentration

INTRODUCTION

Pesticides are the substances or mixture of substances that are widely used in agriculture to protect the plants from pests, weeds and diseases, thereby increasing crop output and preserving food security. They are also employed in public health programmers to protect people against vector-borne diseases such as malaria and dengue fever. Pesticides are required to achieve high yields of agricultural products, but if not managed properly, they might result in pesticide residues in food and environment. Pesticide residues have the potential to contaminate environmental factors such as air, water and soil.

Animals raised for human consumption may absorb pesticides from residues in their feed, water or during direct/indirect pest management measures. Poultry feed and cattle/buffalo feed are the primary source of contamination in chicken, eggs and milk. Due to the lipophilic nature of these pesticides, milk and other fat-rich substances are the key items for their accumulation.

Milk is widely recognized as a balanced diet and a vital source of nourishment for humans. It contains proteins, vitamins, lipids and important minerals that aid in everyday activities and aid in the elimination of malnutrition. The primary source of milk contamination is veterinary medicine given to livestock to kill pests found on the bodies of domestic animals. The most difficult aspect of determining pesticide residues by analytical methods is milk's high protein and fat matrix as interfering compounds. To extract residual pesticides from the milk-involved matrix, pre-treatment and sample extraction are required [1].

Organochlorine Pesticides (OCPs) are a class of chlorinated chemicals. They are introduced by DDT and its isomers (4-4-DDE, 4-4-DDD, etc.), Cyclodienes (Aldrin, Dieldrin, Endrin, Endosulfan, Endosulfan-sulphate, Heptachlor and Heptachlor epoxide) and BHC. OCPs are used as

herbicides and insecticides all over the world. Organochlorine pesticides are characterized by semi-volatility, prolonged half-life and high lipophilicity due to strong environmental persistence. Therefore, it is critical for public health safety and monitor in animal feed, because OCPs are an important part of the food chain.

Organophosphorus Pesticides (OPPs) are commonly used in agriculture, but some, like malathion, are used to treat scabies, lice and crab lice in humans. OPPs are also employed to manage diseases in farm production, veterinary medicine and public hygiene. Unlike OCPs, OPPs have a brief lifetime in the environment and are hence not persistent. Parathion, Diazinon and Malathion are the common examples of organophosphates.

Pesticide usage in agriculture has increased crop output, but unregulated or indiscriminate pesticide use has produced several environmental and health concerns. While pesticides benefit farmers and can help to address the world's growing hunger needs, they have long-term negative effects on a variety of organisms, including humans who depend on them, like cattle, insects, pollinators, soil microbes and earthworm species etc.

Pesticides are utilized by more than half of the world's population living in Asia. In pesticide use, India is rated 12th in the world and third in Asia, after China and Turkey.

Gas chromatography, in combination with various detectors such as mass spectrometry, electron capture detector, nitrogen phosphorus detector and flame ionization detector, is sensitive and widely used method for the detection of volatile pesticides. The proposed research work was planned considering the widespread use of organophosphate and organochlorine pesticides in farms and elsewhere and the possibility of their presence in the human food chain mainly in milk [2,3].

LITERATURE REVIEW

Chemicals and standards

OCPs standard including (α-HCH, β-HCH, γ-HCH, δ-HCH, Aldrin, Heptachlor, Heptachlor epoxide, Trans-chlordane, Endosulfan-I, Cis-chlordane, p',p'-DDE, Endrin, Endosulfan-II, Endrin-Ketone, Endrin aldehyde, Dieldrin, Methoxychlor, Endosulfan sulphate, p',p'-DDT and p', p'-DDD) Mix AB # 3 (20 components) in concentration of 2000 µg/ml each in hexane: toluene (1:1), were procured from Restek Corporation, 110 Benner Circle, Bellefonte, PA 16823, United States.

Similarly, Dichlorvos, Mevinophos, Demeton, Ethoprophos, Naled, Phorate, Diazinone, Disulfoton, Methyl Parathion, Ronnel, chlorpyriphos, Fenthion, Trichloronat, Stirifos, Tokuthion, Merphos, Fensulfothion, Bolstar, Azinophos-methyl and Coumaphos organophosphate pesticides mix (20 components) was procured from Accu-Stand Standard Inc., 125 Market Street, New Haven, CT 06513 USA was provided in concentration of 40 µg/mL, in Hexane: Acetone (95:5).

Organochlorine pesticide standard were further diluted in ethyl acetate and organophosphate pesticide standard, were further diluted in Hexane: Acetone (95:5) to make stock, intermediate and working solutions.

Ethyl acetate, acetone, hexane and Water of HPLC grade and sodium chloride, anhydrous sodium sulphate, anhydrous magnesium sulphate, trisodium citrate of A.R. grade was procured from Advent Chembio private limited Navi Mumbai, Maharashtra 400701, India. disodium citrate of A.R. grade was procured from Sigma Aldrich, Co. 3050 Spruce St, St. Louis, MO 63103, United States.

Sampling

A total of 70 milk samples (200 ml) were collected from local markets in seven target areas name as SA1 to SA7 (Jabalpur, Patan, Sahpura, Kundam, Panagar, Majholi and Sehora) of Jabalpur district, Madhya Pradesh, India, in glass vial. After sampling, samples were stored at -20 °C until further analysis [4].

Extraction of analyte from samples

The extraction method was carried out according to the QuEChERS method with some modifications. For the extraction of OCPs and OPPs in milk sample, a total of 10 g of sample was homogenised and transferred to a 50-ml polypropylene tube.

Then, 10 ml of ethyl acetate was added and shaken vigorously for 1 minute and add 4 g of anhydrous magnesium sulphate and 1 g of sodium chloride to the sample. Afterwards, the sample was thoroughly mixed by a vortex mixer (REMI CM 101 Plus) for 5 minutes.

After that, add 1 g of trisodium citrate and 500 mg of disodium citrate and vortex it for 1 minute and later centrifuged (Remi CPR-24 Plus) at 5000 rpm for 5 min. 2 ml of supernatant was filtered through C-18 cartridge (Isolute, 220-0020-C) with SPE (Biotage® VacMaster™ 20 Sample Processing Station) and transferred to another 3 ml tube with 150 mg of anhydrous sodium sulphate and vortex for 1 min at high speed.

After that, the supernatant was centrifuged (Thermo Fisher Legend X1 R) at 1000 rpm for 5 minutes and 1 ml supernatant was filtered through syringe filters (Nexelo syringe filters, nylon 13 mm, 0.22 µm). The aliquot of extract was then transferred to 2 ml sampling vials for GC-MS analysis.

By using freezing centrifugation, the fatty co-extracted interferents can be easily removed. Since fatty molecules, primarily lipids, have lower melting points than the solvent, they can be removed by centrifugation or filtering, while OCPs stay dissolved in the solvent at varying degrees of freezing temperatures.

Instrumentation conditions

OCPs and OPPs component determinations from target milk samples were performed using a thermo-scientific TRACE 1310 gas chromatograph equipped with an ISQ 7000 mass spectrometer (Thermo Fisher) and the capillary column DB-5MS (5% diphenyl, 95% dimethylpolysiloxane) with 30 m × 0.25 mm i.d., 0.25 µm film thickness (Agilent J and W Scientific Products, Santa Clara, California, USA).The oven temperature programme was as follows: initial temperature of 90°C, held for 0.00 minute; 10°C per minute to 160°C, held for 0.00 min; 5°C per minute to 260°C, held for 0.00 min; 15°C per minute to 280°C, held for 4 minute. The total run time was 32.40 minutes. Injections were performed in a split less mode with an initial injection temperature of 90°C and an injection volume of 1 µL.

The temperature of the ion source was 300°C and the transfer line temperature was 290°C. Chemical ionizations was set to 70 eV and the carrier gas was helium of 99.9999% purity with a flow rate of 1.2 mL/min [5].

The GC-MS was routinely programmed in selective ion monitoring (SIM) mode for residual pesticide analysis, employing three ions. The presence of corresponding target ions and the retention time confirmed the insecticide's presence. After acquisition of the total ion chromatogram for the mixed stock standard solutions of OCPs and OPPs in full scan mode, peaks were identified by their retention time and mass spectra. The target ion

abundances were obtained by injecting OCPs and OPPs pesticide standards under the same chromatographic circumstances, but full-scan conditions were used to identify distinct components of OCPs and OPPs, with the mass/charge scan range from 40 to 550 m/z.

DISCUSSION

Method validation

The proposed method was validated by evaluating the various parameters according to the Nº SANTE/11312/2021 (EC, 2021) guidelines with validation parameters include accuracy, precision, selectivity, sensitivity and reproducibility [6].

Linearity

Working standard solutions of six different concentrations in the range of 10 ppb to 200 ppb for OCPs and OPPs were prepared from standard stock solutions. The standard mix was injected into the GCMS and linear curve was observed from three consecutive injections of each solution. The coefficient values (r^2) were 0.97 to 0.99 for OCPs and 0.90 to 0.99 for OPPs. The representative chromatogram after injecting the standard mixture containing 20 components of OCPs and OPPs.

Specificity

To assess the specificity of the approach total twenty, including ten each of OCPs and OPPs were tested for any interfering matrix signals in the chromatograms.

Recovery

According to the guidance document of Nº SANTE/11312/2021 (EC, 2021), mean recoveries from initial validation should be within the range 70%-120% with an associated repeatability for all analyses within the scope of a method. Recovery investigations were carried out at three different fortification concentrations in the milk sample, as 0.01 mg/kg, 0.05 mg/kg and 0.1 mg/kg of OCPs and OPPs.
The results showed that recovery rates in terms of the mean concentration of six replicates of each spiked concentration ranged between 70% to 110%, with majority of recoveries being greater than 80%. According to the results, the recovery rates for selected pesticides were within an acceptable limit under SENTE recommendations.

Precision

The precision of the approach was assessed in two stages: repeatability (intra-day precision) and intermediate precision (inter-day precision). The RSD of the data from six replicates examined on the same day by the same analyst using the same instrument was used to express intra-day precision. In this study, the intra-day RSDs were in the range of 1.07 to 12.09% for OCPs and 0.32 to 6.85% for OPPs. The RSD of the outcomes of 18 analyses conducted on 3 different days, 6 analyses per day, by the same analyst using the same equipment, served as an indicator of the inter-day precision. In this experiment interday study, RSDs were obtained in the range of 1.40 to 17.98% for OCPs and 4.47 to 17.88% for OPPs obtained using GC-MS [7].

Determination of the Limit of Detection (LOD) and Limit of Quantification (LOQ)

The LOD was in the range of 3.61 to 9.83 µg/kg and 9.89 to 164.14 µg/kg for different components of OCPs and OPPs, respectively. The LOD was estimated as the concentration equivalent to three times the standard deviation of the standard sample of pesticides divided by the slope of the calibration curve. Similarly, for different components of OCPs and OPPs, LOQ was determined as the concentration equivalent to ten times the standard deviation of the pesticide standard sample divided by the slope of the calibration curve was in the range of 10.93 to 29.77 µg/kg and 29 to 497 µg/kg, respectively. The following formulas were used to determine Limits of Detection (LOD) and Limits of Quantification (LOQ): $LOQ=108\delta/S$ and $LOD=3.38\delta/S$, where δ is the standard deviation and S is the slope of the standard curve.

Human health risks assessment

The health risk assessment is necessary to determine the health concerns associated with consumers' dietary exposure to the pesticide content in milk samples. To establish the degree of danger caused by the pesticide residues found in milk samples, an exposure evaluation was undertaken based on monitoring the results of the pesticide residues. For the same, estimated average daily intake and health risk index is calculated. The estimated average daily intake of pesticides was used to calculate the long-term health risk associated with contaminated food consumption (Adeleye et al., 2019), based on Indian adult's average body weights of 65 kg.

The Estimated Average Daily Intake (EADI) was calculated by multiplying the residual pesticide concentrations of each OCPs and OPPs (µg/g) by the milk consumption rate (kg/day) and dividing by body weight. The exposure was calculated on the basis of the per-capita availability of milk (616 g/person/day) in Madhya Pradesh, India (NDDB, 2022) and adults average body weight of 65 kg.

$$EADI(mg/kg/day)=(F(kg/day)\times Cr(\mu g/g))/(Mean\ body\ weight(kg))$$

where F=food (milk) consumption data and Cr is the concentration of the residue in the food sample.
The health risk index (HRI), which was calculated by dividing the EADI by their corresponding ADI values. It was also thought that the rates of absorption and bioavailability were 100%. When the health risk index is greater than one, the food is deemed a risk to consumers; when the index is less than one, the meal is regarded acceptable. where, ADI represents acceptable daily intake from WHO/FAO.

$$Health\ Risk\ Index\ (HRI)=(Estimated\ Average\ Daily\ Intake\ (mg/\ kg/\ kg\ ))/(Average\ Daily\ Intake(mg\ /kg/day))$$

The results of the health risk assessment of the study reveal that the HRI values of OCPs and OPPs were less than one in milk samples, indicating that they are not harmful to humans. Monitoring of the pesticide residue data in milk has been performed. Therefore, monitoring the residues of pesticides in milk continuously is necessary because of possible health effects, widespread uses and insufficient residue data. Additionally, monitoring studies must be performed to improve food safety.

Detection of OCPs and OPPs residues in bovine milk samples

OCPs and OPPs were examined under the circumstances outlined in Section 2.4. The analytes were recognized in the full scan mode with mass range of 45 to 500; and the quantification was carried out in SIM mode in accordance with their retention times, quantification ions (target ions) and qualifier ions.

A total of 70 milk samples, 35 for each OCP and OPPs were analyzed for the determination of twenty components each of OCPs and OPPs and the obtained mean residue levels in milk.

The residue analysis showed that approximately 11.43% (4/35) of the milk samples were contaminated with endosulfan-I and endosulfan-II, heptachlor, α-BHC and δ-BHC. The contamination percentage for the milk samples were 22.86% (8/35), 17.14% (6/35), 14.29% (5/35), 8.57% (3/35) and 5.71% (2/35) for p, p'-DDD, methoxychlor, endrin, heptachlor epoxide and p, p-DDE, respectively. None of the milk samples were found to be contaminated with β-BHC, γ-BHC, aldrin, cis-Chlordane, trans-Chlordane, dieldrin, endrin aldehyde, endosulfan sulphate, p, p'-DDT and endrin ketone.

In the present study, the detected mean residual concentrations of α-BHC in SA1 and SA2 were 2.48±0.07 and 5.41 ± 0.94 µg/kg, respectively, which are lower than those detected by Pandit et al. (2002), who observed the mean residual concentration of α-BHC as 0.012 mg/kg.

Nida (2009) reported that the residual mean concentrations of both α-BHC and endosulfan-I in milk samples were 0.03 mg/kg, which were higher than the mean residual concentration detected in our study.

Maurya et al., collected bovine milk samples from Paliakalan, Uttar Pradesh, India, observed 121 ppb HCH in cow and 149 ppb in buffalo milk sample. Similarly, Muhammad et al., reported presence of α-BHC residues in milk samples with a mean residual concentration of 17.44 ± 3.99 mg/kg [8].

This study revealed that only 11.43 % of samples were positive for α-BHC and 11.43 % were above MRL level according to WHO/FAO; and none of the samples were above MRL level as per the standards of European Union.

Likewise, Wanjiku observed the residue levels of δ-BHC, cis-chlordane, heptachlor, endosulfan-II and p, p'-DDT in cow milk as 0.12, 0.01, 1.723, 2.96 and 3.72 ppb and reported that none samples surpassed the MRL established by Codex Alimentarius, European Union (EU) and USDA.

Oyekunle et al., noticed heptachlor epoxide residual quantities in six different popularly eaten evaporated milk samples from Nigeria. They determined that the residual concentration of heptachlor epoxide (0.374 mg/kg) was higher than the EU's MRL limits. Al-Hawadi (2020) also reported the residues of heptachlor-epoxide in two (6.7 %) milk samples with a mean residual concentration of 0.019 mg/kg.

Lobato et al. detected the residual concentration of endosulfan-I and p, p'-DDE in milk sample ranging between 0.991 to 0.995 mg/kg and 0.991 to 0.995 mg/kg respectively. Shaker et al. (2015) determined the residue levels of dieldrin and methoxychlor in milk samples and showed the presence of dieldrin and methoxychlor in concentration of 0.014 ± 0.001 and 0.142 ± 0.02 mg/kg, respectively in all fifteen collected samples from three different zones of Assiut city.

Ishaq and Nawaz (2018) observed that the residual mean concentrations of dieldrin, p, p'-DDE, endosulfan-II, endosulfan sulfate and p, p'-DDT in milk samples were 11.82 µg/kg, 3.13 µg/kg, 107.16 µg/kg, 91.3 µg/kg, 4.12 µg/kg respectively. Out of which dieldrin and endosulfan-II were observed above the MRL level; and p, p'-DDE, endosulfan sulfate and p, p'-DDT were observed below the MRL level. Hasan et al., also reported that the residual mean concentration of p, p'-DDD and p, p'-DDT in milk samples were 0.09 µg/kg and 19.65 µg/kg in milk samples respectively.

Karakaş and Coşkun investigated 18 different OCPs, viz., aldrin, 4,4-DDD, 4,4-DDE, 4,4-DDT, dieldrin, α-endosulfan, endosulfan sulphate salt, endrin, endrin aldehyde, ketone, heptachlor, methoxychlor, alpha, beta, gamma and delta-HCH in 60 UHT (Ultra Heat Treatment) and 27 pasteurised milk samples using GC-MS/MS and did not find any insecticide residues.

In present study the OPPs pesticide residues detected in raw bovine milk sampled from seven different sample areas of the Jabalpur district. The residue analysis showed that approximately 25.71% (9/35), 28.57% (10/35), 20.00% (7/35), 31.43% (11/35) and 51.43% (18/35) of the milk samples were contaminated with mevinphos, ethoprophos, azinphos-methyl, chlorpyrifos and coumaphos, respectively. None of the milk samples were found to be contaminated with dichlorvos, demeton, naled, phorate, diazinone, disulfoton, methyl parathion, ronnel, fenthion, trichloronat, stirifos, tokuthion, merphos, fensulfothion and bolstar.

The mean concentrations (µg/kg) of mevinphos observed were 21.28 ± 0.27 µg/kg (SA-4), 22.50 ± 0.26 µg/kg (SA-5), 30.36 ± 0.26 µg/kg (SA-6) and 13.88±0.08 µg/kg (SA-7). Similarly, Salas et al. (2003), observed the residue mean concentration of mevinphos as 0.0162 mg/kg in milk samples.

In the current study, the mean concentrations (µg/kg) of ethoprophos were 29.97 ± 0.02 (SA-1), 36.40 ± 0.08 (SA-2), 40.86 ± 0.00 (SA-3) and 30.15 ± 0.16 (SA-4) µg/kg in samples from different sampling areas of the Jabalpur district. These levels were higher than the MRL (0.01 mg/kg) set by the WHO/FAO and USDA and higher than those measured by Yang et al., who detected mean concentrations of 0.006 mg/kg.

In the present study the mean concentrations (µg/kg) of chlorpyrifos were 21.50 ± 0.17 (SA-1), 18.70 ± 0.21 (SA-2) and 93.13 ± 0.04 (SA-7). Similarly, Salas et al., determined the residue levels of chlorpyrifos with mean concentration of 0.0090 mg/kg in collected milk samples. Shaker et al. (2015) also evaluated the chlorpyrifos residue levels and revealed the presence of chlorpyrifos (3.01 ± 1.0 mg/kg) in five out of fifteen milk samples. Dhangar and Patil also detected pesticide residues in buffalo milk sample and reported that the mean residual concentration of chlorpyrifos were between 0.79 and 0.91 µg/mL. Pagliuca et al., also conducted a study to determine OPPs pesticide residues in Italian raw milk and found that the residual mean concentration of chlorpyrifos was 5 µg/kg.

In the present study, the mean concentration of merphos were 5.73 ± 0.36 µg/kg, 1.17 ± 1.40 µg/kg, 63.19 ± 0.04 µg/kg, 34.43 ± 0.27 µg/kg, 35.52 ± 0.11 µg/kg, 65.17 ± 0.46 µg/kg and 30.77 ± 0.07 µg/kg respectively detected in sample areas SA-1 to SA-7.

In current investigation, the mean concentration (µg/kg) of azinphos-methyl was 3.02 ± 1.32 (SA-2),18.35 ± 0.04 (SA-3), 12.21 ± 0.14 (SA-4), 28.48 ± 0.38 (SA-5), 12.94 ± 0.23 (SA-6) and 15.24 ± 0.24 (SA-7) in milk samples. These levels were higher than the MRL (0.01 mg/kg) set by the European Union.

Our results showed that the detected mean residual concentration of coumaphos was 29.99 ± 0.38 µg/kg, 3.33 ± 1.44 µg/kg, 11.07 ± 0.05 µg/kg, 61.36 ± 0.43 µg/kg and 35.11 ± 0.24 µg/kg in the milk sample areas SA-1, SA-2, SA-3, SA-6 and SA-7, respectively.

The OCPs and some OPPs residues found in the samples of bovine milk could be the result of the current or previous application of these pesticides in agricultural fields. This investigation revealed that the pesticides used in the field have not completely vanished; rather, remnants are present in the environment. This may be due to contaminated feed, feed ingredients or agricultural goods used for animal feeding and it may also come from

the water given to the animals. Since the bulk of ingredients used to make feed come from various plant and animal sources, contamination is a possibility at every step, from harvesting raw materials to making the feed. To keep insects away, certain insecticides are used in animal houses, which may enter through inhalation or direct contact with the skin. Over a long period of time, they accumulate in the bodies of animals and may enter the food chain through animal products like milk, meat and eggs [9,10].

CONCLUSION

Following an optimization phase, the QuEChERS technique was used for the determination of OCPs and OPPs in bovine milk using GC-MS. For both OCPs and OPPs, validation study presented acceptable accuracy and precision with recoveries ranging from 70% to 120% and RSD <20%. Compounds showed linear response and determination coefficient (r^2) values>0.99. Method detection limits were ranged from 3.61 to 9.83 µg/kg and 9.89 to 164.14 µg/kg for different components of OCPs and OPPs, respectively; and method quantification limits were ranged from 0.93 to 29.77 µg/kg and 29 to 497 µg/kg, for different components of OCPs and OPPs, respectively. In this research work in milk samples, a large number of OCP residues like α-BHC, β-BHC, γ-BHC, δ-BHC, heptachlor, endrin, endosulfan-I, endosulfan-II and methoxychlor, etc. were detected. Among the OPP compounds, mevinophos, demeton, chlorpyriphos, tokuthion, coumaphos, etc. were found in the collected milk samples. Moreover, two milk samples for demeton and one milk sample for chlorpyrifos were observed to possess the residual concentration above the MRL level.

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