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EFFECT OF DIFFERENT HEAT TREATMENT METHODS ON REDUCTION OF ENDOSULFAN RESIDUES IN MILK OBTAINED FROM DIFFERENT PRODUCTION SYSTEMS IN CHITTOOR DISTRICT OF ANDHRAPRADESH

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Abstract: A study was carried out to know the effect of heat on reduction of Endosulfan pesticide residues in milk collected from different production systems, when subjected for different heat treatment methods. 36 pooled milk samples were collected from Small holders production system, Organized dairy farms, Cooperative societies and Commercial dairy plants collection system. in a randomized block design and fortified with Endosulfan 35% EC at the rate of 0.04mg per kg level. 24 fortified samples were subjected for heat treatments namely Thermization, Pasteurization, Boiling and Sterilization. 6 positive control samples and 6 negative control samples were also analysed for Endosulfan residues. The mean quantity recovery of Endosulfan from milk samples subjected for Thermization, Pasteurization, Boiling and Sterilization were 0.0043±0.00004, 0.0038±0.00004, 0.0025±0.00004 and 0.0023±0.00004 respectively. While, the recovery from positive and negative controls were 0.0330±0.00007 and Nil. The percent recovery was 10.90±0.096, 9.62±0.096, 6.25±0.134 and 5.84±0.094, respectively where as in the positive control it was 82.5±1.82. No traces of Endosulfan were found in the negative control. Among the different methods of heat treatment, sterilization was the most effective followed by boiling and then pasteurization. The least effective method was thermization. There was a significant ($P<0.01$) variation observed for the mean quantity recovery and per cent recovery of Endosulfan residues among different heat treatment methods under evaluation.

Keywords: Heat treatment, Thermization, Pasteurization, Sterilization, Boiling, Fortification, Endosulfan residues, Gas Chromatography.

Introduction

Milk is the important food for all categories of human beings, hence production of clean milk is essential to safe guard the health of the consumers. Endosulfan was the important

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Organochlorine pesticide being widely used in increasing the crop yield. The agricultural waste such as straws and stovers were commonly fed to dairy animals. The residues of Endosulfan are excreted through milk and when such milk is consumed by the humans may lead to toxicological manifestations. A study was undertaken to know the effect of different heat treatment methods in reducing the residues of Endosulfan in milk and to evaluate the effectiveness of these methods.

Materials and methods

Thirty six (36) pooled milk samples were collected in a Randomized Block Design (RBD) from four production systems ie small holders, organized dairsy farms, cooperative societies and commercial dairy plants collection system and subjected for four heat treatment methods like, Thermization, Pasteurization, Boiling and Sterilization (6 x 4 = 24 samples) and six (6) samples were used as Positive control and another six (6) samples as Negative control. All the samples were fortified with 0.04 ppm of Endosulfan. The fortified Milk samples were analysed by Gas Chromatography in the Bio-chemistry laboratory of the College of Veterinary Science, Tirupati.

Analytical method: Multi-residue analysis as prescribed by (AOAC, 1995) was followed for determination of Endosulfan residues in milk samples which includes: **Extraction**: The residues of Endosulfan in milk were extracted with Di Ethyl Ether and Hexane (1:1).**Partitioning and Clean –up** was done by means of Florisil chromatographic column of internal diameter of 1.5 mm and length 45 cm the extract was eluted with Dichloro Methane and Hexane (1:1). **Concentration**: the extract was concentrated and made the volume to 5ml with Hexane for GC analysis. **GC analysis**: The GC equipment used for analysis was of Varion CP 3800 and Shimadzu 2010 equipped with Electron Capture Detector. The Limit of detection was 0.0003 ppm and the Limit of quantitation was 0.001 ppm.

Chromatograms of Standard and Samples: Endosulfan standard at 0.1 ppm level was injected into the Gas Chromatograph equipment, The chromatogram was plotted graphically taking the retention time on ‘X’ axis and electric current in terms of volts / milli volts on ‘Y’axis .The Endosulfan standard had shown peak area at a retention time of 15.871 min. The standard was used to determine the levels of Endosulfan residues in the milk samples by comparing the recovery of Endosulfan residues in the standard and sample. Peak area was used to determine the quantity of Organo chlorine compound in the sample.

Endosulfan standard solution: A 0.1 ppm of Endosulfan working standard was prepared from commercial Endosulfan 35% EC ie. Hysulfan, manufactured by HYDERABAD CHEMICALS LIMITED, A-24/25, A.P.I.E. Balanagar, Hyderabad-500037.

Fortification of milk samples: The milk samples were divided into 6 sets of 6 samples each and each sample of milk was fortified with Endosulfan at the rate of 0.04 mg / kg. The first set served as a negative control which was neither fortified nor subjected for any of the heat treatment methods where as the 2^{nd} set of milk samples were served as positive control which was fortified but not subjected for any of the heat treatment methods and analysed for Endosulfan residues. Set-3 to Set-6 were subjected for Thermization, Pasteurization, Boiling and Sterilization. The samples were cooled and analysed by GC-ECD. After obtaining the chromatograms the recovery per cent of the Endosulfan, was compared with positive control to find out the efficiency of the heat treatment methods on the reduction of residual levels of Endosulfan.

Heat treatment methods

a. Thermization: The fortified milk samples were heated to 63^0c with no holding time cooled and analysed for Endosulfan residues by GC-ECD.

b. Pasteurization: The fortified milk samples were heated to 72^0C for 15 seconds and allowed to cool it, then 500 ml was filled in the container and analysed for Endosulfan residues by GC-ECD.

c. Boiling: The fortified milk samples were subjected to boiling and kept for 5mts, cooled and analysed for Endosulfan residues by GC-ECD

d. Sterilization: The fortified milk samples were heated to 120^0C for 15 mts at 15 psi pressure then cooled and analysed for Endosulfan by GC-ECD.

ANOVA was done to findout significant difference among different heat treatment methods with respect to quantity and percent recovery as per the methods of Snedecor and Cochran (1994).

Results and discussion

The data on quantity recovery and per cent recovery of endosulfan residues was presented in Table 1 and graphical representation in Figure 1. A significant variation ($P<0.01$) was observed among different heat treatment methods. Sterilization was found to be most effective with least quantity recovery and per cent recovery while Thermization was most recovered and most percent recovered and hence least effective. It was found that the heat

treatment methods Boiling and Sterilization had shown a significant (P<0.01) difference in reducing the Endosulfan residues when compared to Pasteurization and Thermization.

The results obtained in the present study were similar to those obtained by Rabo *et al.* (1989) Ali *et al.* (1993) and Abdrabo *et al.* (1989) in DDT and other pesticides. Mitchell *et al.* (1986) found isomers of BHC residues in pasteurized milk samples from queensland to the extent of 26.6, 69.2 and 17.5 per cent in α-BHC, β-BHC, γ-BHC, respectively. Similarly Maitre *et al.* (1994) determined heptachlor and its epoxide (98%), α and γ-isomer of HCH in (88 and 84% samples) Aldrin and Dieldrin, Chloridae (α and γ)), Endosulfan I and II, DDT isomer and HCB in (69, 77, 41, 49 and 17 %), respectively in pasteurized milk purchased from super market in Santa Fe and other Argentinian cities. while, Liska (1968) monitored the effect of different processing methods like drying and sterilization of milk which destroyed some of the residues present. He also mentioned that the amount of residue destroyed was varied with processing treatment and nature of insecticide residue. Rajasekhar *et al.* (2007) reported that the greater loss of α and β endosulfan (13.11 and 27.6 per cent) were found when milk was subjected for sterilization.

Conclusion

Among the different heat treatment methods, Sterilization was found to be most effective with least quantity recovery and per cent recovery while Thermization was most recovered and most percent recovered and hence least effective. Thermization and Pasteurization varied significantly (P<0.01) with Boiling and sterilization. It was found that the heat treatment methods Boiling and Sterilization had shown a significant (P<0.01) difference in reducing the Endosulfan residues when compared to Pasteurization and Thermization. In the present study Boiling and Sterilization were equally good in reducing the levels of Endosulfan and can be suggested for improving the quality of milk for processing.

Acknowledgements

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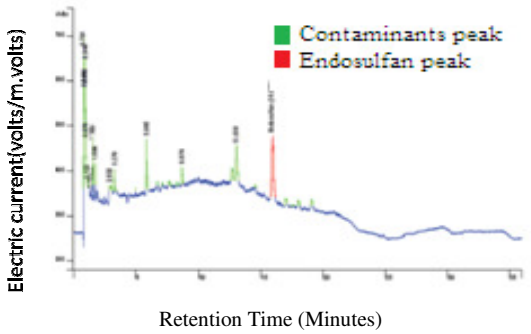
Table 1: Effect of different Heat treatment methods on reduction of Endosulfan residues in milk fortified with Endosulfan (n=36)

Sl. No.	Method of heat treatment	Level of fortification (ppm)	Quantity recovered (ppm) Mean+SE	Recovery (%) Mean+SE	Maximum Residue Limit (ppm)	
					On milk basis	On 4% Fat basis
1.	Thermization	0.04	0.0043 ± 0.00004^{b}	10.90 ± 0.096^{b}	0.004	0.1
2.	Pasteurization	0.04	0.0038 ± 0.00004^{b}	9.62 ± 0.096^{b}	0.004	0.1
3.	Boiling	0.04	0.0025 ± 0.00004^{a}	6.25 ± 0.134^{a}	0.004	0.1
4.	Sterilization	0.04	0.0023 ± 0.00004^{a}	5.84 ± 0.094^{a}	0.004	0.1
5.	Positive control	0.04	0.0330 ± 0.00007^{c}	82.5 ± 1.82^{c}	0.004	0.1
6.	Negative control	--	--	--	0.004	0.1
7.	Mean ±SE	--	0.00918±E0.00596	23.022±14.900	0.004	0.1

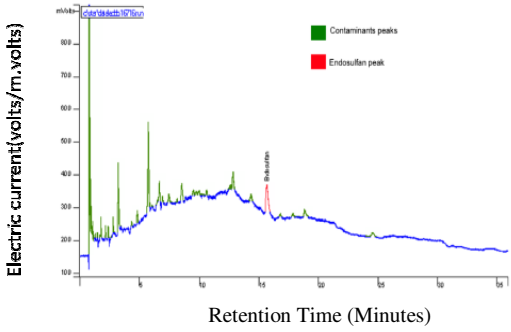
*Values with same superscripts in the row wise do not differ significantly ($P < 0.01$)
**MRL: Maximum Residue Limit
***BDL: Below Detectable Limit of 0.0003ppm.

Figure: GC-ECD Chromatograms of milk samples subjected for different heat treatments methods

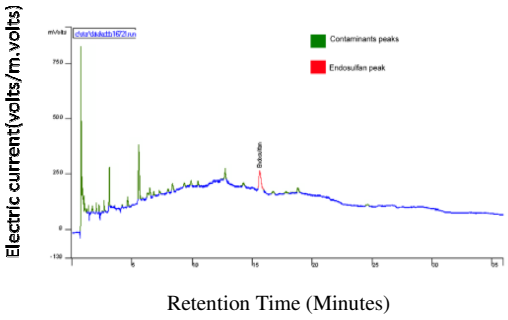
Endosulfan Standard



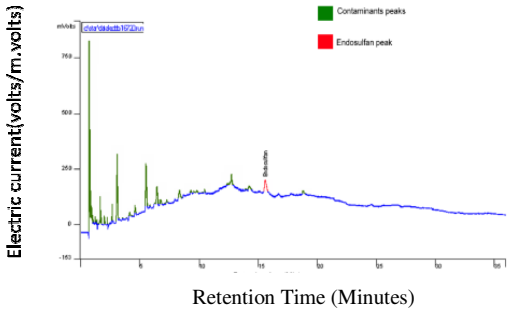
Thermization



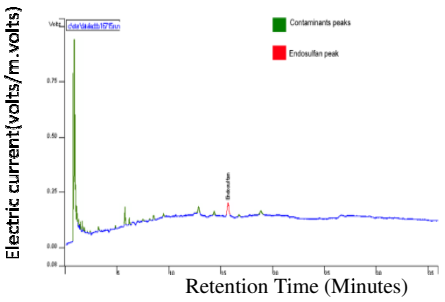
Pasteurization



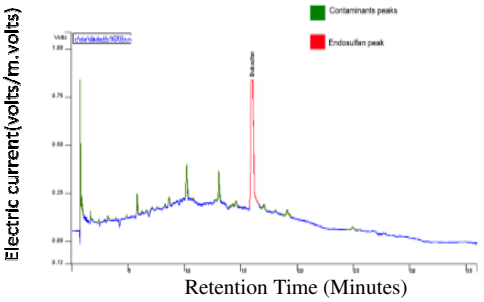
Boiling



Sterilization



Positive Control



Negative Control