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Application of in-plant control measures in some Egyptian micro-scale dairy enterprises and its impact on heavy metal contents of their products

Magda Magdy Abd El-Salam · Mohamed F. Farahat · Gaber I. Abu-Zuid · Samia G. Saad

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Abstract Egypt is encouraging micro-scale enterprises as proved to be one of the most important reasons of economic growth. Most of the annual milk production is processed in micro-scale dairy enterprises located in squatter areas with high health risks and negative environmental impact. The aim of this study was to assess the effectiveness of in-plant control measures in controlling lead and cadmium levels in dairy products from nine Egyptian micro-scale enterprises. The results revealed that white cheese enterprises had the highest mean lead and cadmium contents; both in their raw milk (0.712 and 0.134 mg/L, respectively) and final products (0.419 and 0.061 mg/kg). Higher compliance percentages were found with cadmium levels specified in the Egyptian standards than with lead levels and ranged from 59.4% in raw milk to 100% in dry milk for cadmium levels and from 8.3% in white cheese to 66.7% in ice cream for lead; moreover, none of the collected raw milk samples were complying with the lead levels. After implementation of in-plant control measures, lower lead levels were found in all samples with reduction percentages ranging from 35.2% in raw milk from the ice cream enterprises to 73.2% in yoghurt; moreover, higher percentages of samples complied with cadmium levels. This study highlights the urgent need for applying in-plant control measures to the Egyptian micro-scale dairy enterprises to improve both safety and quality of their products.

Keywords Heavy metals · Dairy products · Micro-scale enterprises · In-plant control

M. M. Abd El-Salam (✉)
Biology Department, College of Science and Humanity Studies, Prince Sattam bin Abdulaziz University, Al-Kharj, Kingdom of Saudi Arabia
e-mail: mmagdy_hiph@yahoo.com

M. M. Abd El-Salam · G. I. Abu-Zuid · S. G. Saad
Environmental Health Department, High Institute of Public Health, Alexandria University, Alexandria, Egypt

M. F. Farahat
Nutrition Department, High Institute of Public Health, Alexandria University, Alexandria, Egypt

M. F. Farahat
Community Health Sciences Department, College of Applied Medical Sciences, King Saud University, Riyadh, Kingdom of Saudi Arabia

Introduction

Egypt is one of the economically developing countries that are trying to boost their economy through the industrial sector (El-Mahgary and Hamed 2002). Egypt is encouraging micro-scale enterprises as proved to be one of the most important reasons of economic growth as in China and Malaysia (Ministry of International Trade and Industry 1995). In 2003, micro-scale food enterprises in Alexandria comprised 16.4% of the Egyptian food enterprises (Egyptian Ministry of Manpower 2003). About 80% of the annual milk production (2.8 million tons) is processed in micro-scale dairy enterprises located in squatter areas (El-Gendy 2001). Most of them are not licensed, and their products may not

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meet the hygienic requirements. The health risks and environmental impact associated with these enterprises are expected to be higher than in larger-scale well-controlled industries (Egyptian Food Industries Chamber 2002). The use of old technology resulted in several negative environmental impacts and reduced quality of their products (Ministry of International Trade and Industry 1995). Milk and milk products constitute an important component of the human diet especially for growing children. Thus, contamination of milk by heavy metals constitutes a health risk to the human population (Crout et al. 2004). Today, the consumer is more demanding and expects "healthy" milk, rich in nutrients, with high biological value, but without health risks (Licataa et al. 2004). Contamination of milk and its products by heavy metals is one of the major problems confronting public health. Lead and cadmium have received increasing attention due to their adverse toxic effects and raising major concerns in food safety (Saad et al. 2001a, 2001b). Dairy products can become contaminated with heavy metals when the lactating animals are grazing on contaminated fields (UNEP 2002). Chronic poisoning due to intake of smaller quantities of heavy metals leads to their accumulation in tissues, bones, hair, and blood and significantly increased their levels in food of animal origin (Patra et al. 2005; Swarup et al. 2005). Lead and cadmium may be present in the environment either from natural sources or from anthropogenic activities. Other sources include water, equipment, and utensils used during their processing. (Carl 1991). The aim of this study was to assess the effectiveness of in-plant control measures in controlling lead and cadmium levels in dairy products from nine Egyptian micro-scale enterprises.

Material and methods

Study setting and design

In our previous study (Abd El-Salam et al. 2006), 60 micro-scale dairy enterprises were selected by a simple random sampling technique where a hygiene and sanitation survey was conducted using a predesigned questionnaire. These 60 enterprises represented one third of the micro-scale dairy enterprises in rural and semi-urban areas located in El-Beheira and Alexandria governorates, Egypt. For the current study, only 9 out of these 60 enterprises accepted their exposure to further investigation and sample collection since the majority of micro-scale enterprises located in areas where hygienic and sanitary measures are usually not followed. They are known as illegal stairway enterprises and most of their dairy products are not complying with health laws or standards.

The study was carried out in three stages: (1) pre-in-plant control stage with hygiene and sanitation assessment of nine 9 selected micro-scale enterprises (three from each of white cheese, yoghurt, and ice cream micro-scale enterprises), (2) intervention stage on three out of these nine enterprises (one of each of white cheese, yoghurt, and ice cream enterprises) which approved the application of the suggested in-plant control measures, and (3) post-in-plant control stage with reassessment of the three 3 enterprises where in-plant control measures were applied.

Sampling and collection

During pre-in-plant control stage, a total of 72 samples of raw milk (fluid or dry) and final dairy products were collected from the nine selected micro-scale dairy enterprises. The samples were collected monthly for 4 months from each enterprise. During post-in-plant control stage, a total of 24 samples were collected once a month for a period of 4 months. Four samples from each of raw milk and final dairy products were collected from each of three enterprises where in-plant control measures were applied. All test samples were collected and analyzed in triplicate for their lead and cadmium levels.

Sampling preparation and analysis

All samples were digested according to the analytical methods No. 25. 021, No. 25. 056 of the Association of Official Analytical Chemists (AOAC 1995) where 1 g or ml of each sample was placed into a macro-Kjeldahl digestion flask, then 6 ml of concentrated nitric acid and 1 ml of concentrated perchloric acid were added. The mixture was boiled until dense white fumes appeared and the mixture became clear. The digest was diluted to a suitable volume (25 ml) with distilled water after its filtration using Whatman No. 42-μm filter paper.

Lead and cadmium were selected for measurements because they are the most common poison heavy metals in Egyptian dairy products as shown by several studies cited in our discussion. Lead and cadmium were measured against their standard curves using the appropriate absorption standard solution (1010 μg pb/ml in 1.1 HNO_3) or (1000 μg cd/ml in 1.1 HNO_3) using an atomic absorption spectrophotometer, AAS (Shimadzu model AA-6650, Duisburg, DE) flame system. The results were reported on wet weight basis. A blank was made with the same acids and subjected to the same digestion procedures for correction of impurities present in acids, reagent water, and glassware. The detection level for each of lead and cadmium was 0.06 mg/kg. Samples containing lead or cadmium below the detection levels were reported as not detected (ND). All reagents were purchased from Sigma and were of analytical grade. The precision of the applied analytical method was validated through an accurate analysis of the standard reference material (SRM-1549a, WholeMilk Powder, NIST, Gaithersburg, MD, USA).

Statistical analysis

Data was tabulated and presented in a form of arithmetic mean and standard deviation for lead and cadmium using SPSS version 11.0 computer software package (Daniel 1995). The cut-off point for statistical significance was p value < 0.05, and all tests were two-sided. Paired sample t test was carried out to detect the significant difference between the mean lead and cadmium levels before and after application of in-plant control measures.

Results

Hygienic and sanitary conditions before application of in-plant control measures

There were several hygienic and sanitary problems associated with handling of milk and its products encountered in the nine selected micro-scale enterprises before application of in-plant control measures. These problems included using non-cooling vehicles to transport the raw milk except in only one of the white cheese enterprises, storing the fluid milk at room temperature in all white cheese enterprises and in more than half of yoghurt and ice cream enterprises (66.7%). Stainless steel containers were not used during the manufacture of dairy products except in only one white cheese and two of ice cream enterprises, cleaning of equipment once at the end of the working day in all enterprises and warm water with detergents were used in only one of ice cream enterprises, improper storage temperatures of the final products in two-third of these enterprises; moreover, non-cooling vehicles were used to transport the final products in one third of each of yoghurt and ice cream enterprises and in two-thirds of white cheese enterprises.

Hygienic and sanitary conditions after application of in-plant control measures

Hygienic and sanitary conditions of equipment and utensils improved after implementation of the in-plant control measures in the three studied enterprises; handling of milk and its products was also improved with the highest improvement in yoghurt and ice cream enterprises.

Lead and cadmium levels before and after application of in-plant control measures

Heavy metal analysis of 72 samples collected from the nine micro-scale dairy enterprises revealed that the highest mean lead and cadmium levels was in the raw milk of the white cheese enterprises (0.712 and 0.134 mg/L, respectively) and in their final products (0.419 and 0.061 mg/kg, respectively). The lowest lead levels (0.359 mg/L and 0.108 mg/kg) were in the raw milk and final products collected from the ice cream enterprises whereas their cadmium levels were 0.024 mg/L and 0.033 mg/kg, respectively (Table 1).

Compliance of samples collected from the nine micro-scale dairy enterprises with cadmium levels specified in the Egyptian standards was higher than those with lead levels. The compliance percentages with cadmium levels ranged from 59.4% in raw milk to 100% in dry milk compared to compliance percentages with lead ranging from 8.3% in white cheese to 66.7% in ice cream. It is worth mentioning that all the collected raw milk samples violated the permissible limits for lead levels (Fig. 1).

Lead and cadmium levels reduced in all samples collected after implementation of in-plant control measures. Lead reduction percentages ranged from 35.2% in raw milk from the ice cream enterprise to 73.2% in yoghurt although the compliance percentages with lead levels specified in the Egyptian standards increased only in the final products of three studied enterprises. Higher compliance percentages with cadmium levels specified

Table 1 Lead and cadmium levels of milk and final dairy products collected from the nine selected micro-scale enterprises

Micro-scale dairy enterprises	Samples		Lead level (mg/kg or L) ($\overline{X}$ ± SD)	Cadmium level (mg/kg or L) ($\overline{X}$ ± SD)
	Type	No.		
White Cheese	Raw milk	12	0.712 ± 0.455	0.134 ± 0.234
	Final product	12	0.419 ± 0.396	0.061 ± 0.076
Yoghurt	Raw milk	12	0.587 ± 0.349	0.083 ± 0.096
	Final product	12	0.210 ± 0.219	0.040 ± 0.048
Ice cream	Dry milk	4	0.094 ± 0.109	0.019 ± 0.007
	Raw milk	8	0.359 ± 0.139	0.024 ± 0.026
	Final product	12	0.108 ± 0.207	0.033 ± 0.032

in the Egyptian standards were observed with higher reduction percentages except for raw milk collected from the ice cream enterprise (Table 2).

Discussion

Micro-scale dairy enterprises are playing a very important role in the Egyptian economy in terms of employment and as a training ground for entrepreneurs before they invest in larger enterprises. However, they face several constraints because they use old technologies and their products are inconsistent due to lack of quality control facilities, lack of trained workers, and poor manufacturing practices (Egyptian Food Industries Chamber 2002). Therefore, some micro-scale dairy enterprises willing to develop their business were selected to participate in this study where certain in-plant control measures were undertaken to improve their performance.

Hygienic and sanitary conditions before application of in-plant control measures

The results of the present study were inconsistent with the survey results obtained by El Derea et al. (2003) in Alexandria, Egypt which revealed that equipment and utensils were one of the most important sources of

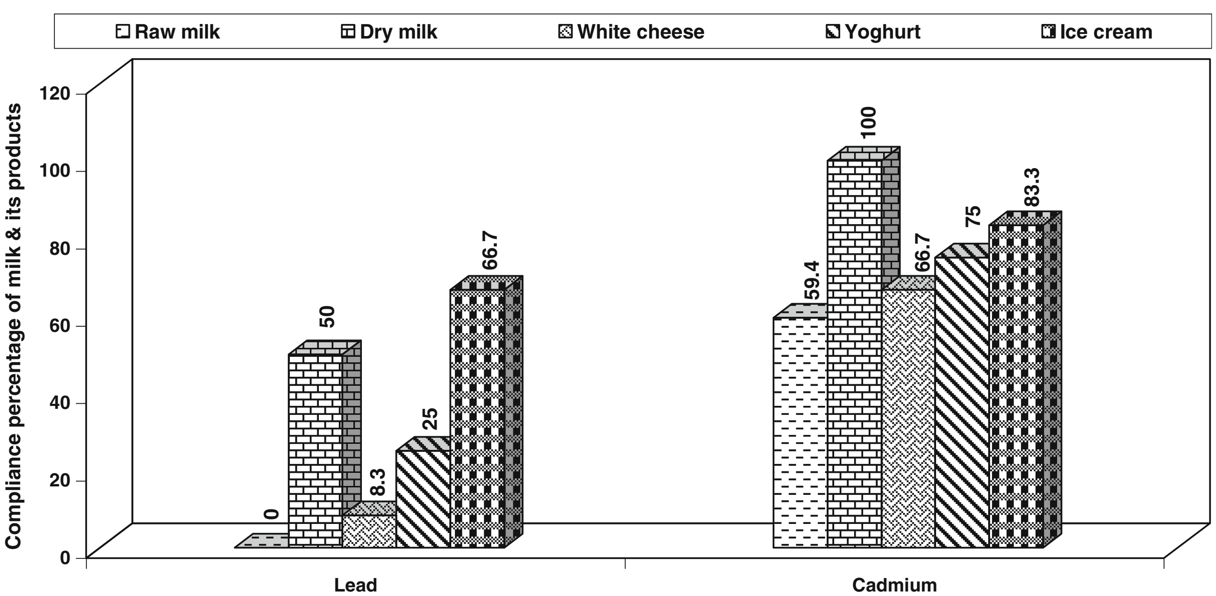

Fig. 1 Compliance percentages of milk and dairy products collected from the nine enterprises with lead and cadmium levels specified in the Egyptian standards

Table 2 Lead and cadmium levels of raw milk and final dairy products collected from the three studied micro-scale dairy enterprises before and after in-plant control

Micro-scale dairy enterprises (N = 3)	Type of samples	No. of samples		Lead level (mg/kg or L)						Cadmium level (mg/kg or L)					
		Before	After	Before		After		%[a]	Paired *t* test	Before		After		%[a]	Paired *t* test
				$\bar{X} \pm SD$	Compliance with standards (%)	$\bar{X} \pm SD$	Compliance with standards (%)			$\bar{X} \pm SD$	Compliance with standards (%)	$\bar{X} \pm SD$	Compliance with standards (%)		
White Cheese	Raw milk	4	4	0.639 ± 0.388	0.0	0.188 ± 0.062	0.0	70.6	1.926*	0.278 ± 0.387	25.0	0.016 ± 0.0047	100.0	94.2	1.926*
	Final product	4	4	0.332 ± 0.074	0.0	0.112 ± 0.095	50.0	66.3	1.926*	0.058 ± 0.080	75.0	0.006 ± 0.0054	100.0	89.7	1.461
Yoghurt	Raw milk	4	4	0.753 ± 0.586	0.0	0.346 ± 0.088	0.0	54.1	1.926*	0.149 ± 0.143	25.0	0.039 ± 0.019	75.0	73.8	1.461
	Final product	4	4	0.235 ± 0.375	50.0	0.063 ± 0.084	75.0	73.2	1.069	0.058 ± 0.079	75.0	0.015 ± 0.011	100.0	74.1	1.604
Ice cream	Raw milk	4	4	0.335 ± 0.143	0.0	0.217 ± 0.053	0.0	35.2	1.461	0.014 ± 0.011	100.0	0.010 ± 0.0069	100.0	28.6	0.535
	Final product	4	4	0.30 ± 0.286	0.0	0.081 ± 0.057	50.0	73.0	1.461	0.069 ± 0.088	50.0	0.001 ± 0.0012	100.0	98.6	1.461

ND not detected

*$p < 0.05$

[a] Reduction percentages in lead and cadmium [(concentration before in-plant control − concentration after in-plant control)/concentration before in-plant control × 100]

contamination of raw milk and its products when they were inadequately cleaned, sanitized, and dried. In California, Quality Assurance Program (2000) stated that equipment and utensils should be maintained in a good condition, free from rust, milk stone, or any unsanitary conditions. Also, they should be washed, rinsed, and drained after each use and sanitized before each use. Middlemiss et al. (1985) reported that the effects of the washing process were dependent upon temperature where about 95% of both protein and fat can be removed at 60 °C but only about 80% of protein and 60% of fat at 20 °C.

The current study was in compliance with the Western Australian Regulation which specifies that raw milk should be cooled and stored in a cooling tank at below 5 °C (Bell and Gallagher 1999). Also, our results were incompatible with the Pennsylvania Department of Agriculture (1982) which specified that all multiuse containers, equipment, and utensils exposed to milk or milk products should be made of smooth impervious, non-absorbent, corrosion-resistant, non-toxic, heat-resistant, and safe materials such as stainless steel. However, similar results were obtained by Ramadan et al. (1998), who found that the majority of the storage containers used in dairy plants located in Kafr El-Dawar, Egypt were plastic, barrel, and metal.

Hygienic and sanitary conditions after application of in-plant control measures

Adequate cleaning of equipment and utensils after each use by hot water and detergents instead of using cold water alone after each shift improved sanitation of equipment and utensils in all selected enterprises. In the white cheese enterprise, the wooden rod that was used for stirring the raw milk during its heat treatment was replaced by a manual stainless steel paddle; moreover, the wooden tables and deteriorating cheese gauzes were replaced by metal top tables and new cheese gauzes. In the yoghurt enterprise, the metal pan and rod used during the production process were replaced by a stainless steel vat that was mechanically stirred using a stainless steel paddle and surrounded by a water jacket. In the ice cream enterprise, a cooling tank was used in the aging process and periodical maintenance of its pasteurizer was established to improve the efficiency of the heating process to be maintained at 95 °C for 10 min.

Lowering the milk temperature to < 5 °C during its transportation, storage of the raw milk in a cooling tank until it is processing and using controlled electric incubators instead of using a coal-heated cupboard contributed to the improvements in the investigated yoghurt enterprise. On the other hand, improvement in the investigated white cheese and yoghurt enterprises was carried out by changing the temperature of the heat treatment process to be 73 °C for 15 s and 85 °C for 30 min instead of 45 and 43 °C, respectively. In addition, storing the dry milk used in ice cream manufacturing and refrigerating instead of keeping it in a damp area at room temperature attributed to its product improvement.

Lead and cadmium levels before application of in-plant control measures

Among samples collected from the nine micro-scale dairy enterprises, white cheese had the highest mean lead (0.419 mg/kg) and cadmium (0.061 mg/kg) levels. This can be attributed to the old machinery with lead soldering, low-quality salt obtained from the dried marshes, using cadmium-plated utensils, and leachable cadmium-based pigments in plastic food-containers. Ice cream samples had the lowest lead and cadmium levels since the majority of their enterprises used stainless steel equipment. Another study by Abdou and Korashy (2001) reported higher mean lead levels in Domietta cheese and yoghurt samples (1.53 and 2.7 mg/kg, respectively) and higher cadmium levels in yoghurt samples (0.17 mg/kg), although they reported lower lead levels in ice cream samples (0.078 mg/kg), and cadmium was below the detection level in Domietta cheese and ice cream samples. Also, higher mean lead (0.8 and 1.52 mg/kg) and cadmium (0.5 and 0.22 mg/kg) levels in white cheese were reported by Ghoniem (1985) and El-Okazy (1995), respectively. Ismail (2003) reported that the mean lead levels in white cheese samples varied between 0.197 and 0.759 mg/kg while cadmium levels ranged from 0.028 to 0.259 mg/kg.

In the present study, the mean lead and cadmium levels of raw milk used for white cheese production were higher than those used for yoghurt and ice cream (0.712, 0.587, and 0.359 mg/L, respectively for lead) and (0.134, 0.083, and 0.024 mg/L for cadmium), and this may be attributed to delivering raw milk from different suppliers. Sharkawy and Hussein (2002) reported that lead levels in milk samples collected from three farms ranged from 0.368 to 0.648 mg/L while cadmium levels ranged from 0.053 to 0.169 mg/L. El-Prince and Sharkawy's (1999) study indicated that lead levels in raw milk collected from eight different governmental dairy ranged from 0.123 to 0.384 mg/L while cadmium levels ranged from 0.004 to 0.031 mg/L.

The present study revealed that the mean lead and cadmium levels of raw milk samples were higher than their corresponding final products. This may be attributed to retardation of lead and cadmium absorption by calcium chloride added during cheese making and their removal after binding with whey proteins (FAO/WHO 2000). Moreover, in the case of ice cream, the addition of 55–76% of water during its processing may play a role in decreasing their levels. This finding is consistent with the results obtained by El-Tawila et al. (1998) who found that Domietta cheese samples collected from micro-scale dairy plants had lower lead levels (0.13 to 0.14 mg/kg) than raw milk used for its processing (0.29 mg/kg).

None of the analyzed raw milk collected from the selected nine micro-scale enterprises was in compliance with lead levels specified in the Egyptian standards (0.1 mg/kg) (Egyptian Organization for Standardization 1993) despite its compliance reaching the highest percentage (66.7%) in ice cream. Although the permissible cadmium levels (0.05 mg/kg) are much lower than that of lead (Egyptian Organization for Standardization 1993), cadmium compliance percentages were higher reaching its maximum in dry milk where all samples were complying and this may be attributed to wider lead distribution in the environment. Saad et al. (2001a, 2001b) reported a higher percentage of compliance among Domietta cheese and yoghurt samples where 70% of each was within the permissible limit of lead.

Lead and cadmium levels after application of in-plant control measures

There were noticeable reductions in lead and cadmium levels in all raw milk and final products after implementation of in-plant control measures in the three studied enterprises. The reductions in lead levels were significant ($p < 0.05$) in raw milk collected from white cheese and yoghurt enterprises as well as in white cheese samples, constituting reduction percentages of 70.6, 54.1, and 66.3%, respectively. This can be attributed to soldering of white cheese tins in a separate area away from the white cheese production area, using higher-quality salt in cheese making and replacing the old equipment soldered with lead by stainless steel ones.

Although all samples of raw milk and dairy products showed a reduction in their cadmium levels after implementation of in-plant control, this reduction was significant ($p < 0.05$) only in the case of raw milk collected from white cheese enterprise. Despite, ice cream samples showing the highest mean cadmium level (0.069 mg/kg) before in-plant control, they had the lowest level (0.001 mg/kg) after its implementation with the highest reduction percentage (98.6%). This can be attributed to replacing plastic containers colored with cadmium compounds with stainless steel ones that are devoid of cadmium (Preda et al. 1983; Technocon Engineers 1992).

After implementation of in-plant control measures, the compliance percentages with the Egyptian standards concerning lead level increased to reach 50% in each of white cheese and ice cream samples and to 75% in yoghurt samples. Moreover, all dairy products and milk collected from white cheese and ice cream enterprises were complying with cadmium levels. In Poland, the proportion of dairy samples contaminated with lead and cadmium levels exceeding the highest permissible limits has been decreasing to reach 2% after adoption of the HACCP system (Szponar and Wojton 2002).

Conclusions and recommendations

Although the application of in-plant control measures successfully reduced lead and cadmium levels of raw milk and final dairy products, some of them were still not complying with the Egyptian standards. Lax measures from the responsible authorities can be a driving force for owners of micro-scale dairy enterprises to act in their regular ways of production that lacks quality control measures. So, continuous improvement of conditions under which dairy products are produced is a must and a responsible authority should periodically collect samples to assess their compliance. It is preferable to rely on a system like the hazard analysis and critical control point "HACCP" to ensure the safety of produced dairy products rather than relying on the end-product testing.

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