Gutiérrez Tolentino, Rey; Noa Pérez, Mario; Díaz González, Gilberto; Vega y León, Salvador; González López, Magdalena; Prado Flores, Guadalupe

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Asociación Interciencia
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DETERMINATION OF THE PRESENCE OF 10 ANTIMICROBIAL RESIDUES IN MEXICAN PASTEURIZED MILK

Rey Gutiérrez Tolentino, Mario Noa Pérez, Gilberto Díaz González, Salvador Vega y León, Magdalena González López and Guadalupe Prado Flores

SUMMARY

Residues of 9 antimicrobial agents approved in México for veterinary use in dairy cattle (sulfathiazole, sulfamerazine, sulfamethazine, sulfachloropyridazine, sulfamonomethoxine, sulfamethoxazole, nitrofurazone, furaltadone, and furazolidone) and non-approved chloramphenicol residues were studied every two weeks in four commercial brands of Mexican pasteurized milk (A, B, C and D) during one year (n=4×24=96). Drug residues were analyzed by high pressure liquid chromatography HPLC with UV detection. Percentage of positive samples to sulfonamide residues were 47.2% (Milk A), 58.3% (Milk B), 44.7% (Milk C) and 50% (Milk D). Sulfonamide residues ranged between 1.9 and 180µg/kg. Sulfathiazole and sulfacloropiridazina were the most common residues found. Only three samples (Milk B) exceeded maximum residue levels established for sulfonamides. Nitrofurans residues were not detected in any milk sample. Illegal Chloramphenicol residues were detected in one milk sample (Milk A) in levels of 27.2µg/kg.

KEYWORDS: Antimicrobial Residues / Chloramphenicol / Nitrofurans / Pasteurized Milk / Sulfonamides /

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Introduction

Antimicrobial drugs, such as sulfonamides, nitrofurans and antibiotics, are widely used for treatment and prevention of disease in dairy cattle. Residues of these drugs may therefore be present in milk and can be a health hazard to consumers, considering that they may cause allergic reactions in sensitive persons (Thomson and Sporns, 1995), increase in the number of antibiotic resistant pathogenic bacteria (Honkanen and Reybroeck, 1997) and some of them, like sulfamethazine and nitrofurazone are considered to have carcinogetic properties (Paulson et al., 1992). The monitoring of antimicrobial residues in commercial milk is essential for ensuring the safety and adequacy of food, as well as providing an indication of the proper usage of these chemothapeutics at the producer level (Zomer et al., 1992).

The purpose of this work was to evaluate the residual levels of 9 antimicrobial drugs (6 sulfonamides and 3 nitrofurans) approved for veterinary treatments in dairy cattle, as well as illegal chloramphenicol residues in whole pasteurized commercial milk.

Materials and Methods

Procurement of milk

Three commercial brands with wide national distribution (A, B, C) and one brand (D) produced and distributed by the government stores for the low income population, were selected. Sampling was carried out in supermarkets (A, B, C) and in government stores (D).
Os resíduos de 9 agentes antimicrobianos aprovados no México para uso veterinário no gado leiteiro (sulfatiazol, sulfametazine, sulfadimetoxina, sulfachloropyridazina, sulfamonometoxina, sulfametoxazol, nitrofurazona, furaltadona e furazolidona), assim como de cloranfenicol, cujo uso está proibido na produção pecuária, foram determinados no leite integral pasteurizado e comercializado na Cidade do México. Quatro indústrias de leite (A, B, C e D) foram estudadas quinzenalmente durante um ano (n=4×24=96). Os resíduos foram analisados por cromatografia de líquidos de alta pressão (HPLC) com detector UV. A porcentagem de amostras positivas a resíduos de sulfonamidas foi de 47,2% (leite A); 58,3% (leite B); 44,7% (leite C) e 50% (leite D). Os resíduos de sulfonamidas estiveram no intervalo de 1,9 a 180µg/kg. Resíduos de sulfatiazol e sulfametazina foram os mais encontrados. Somente três amostras (leite B) excederam os níveis de resíduo máximo estabelecidos para sulfonamidas. Não se detectaram resíduos de nitrofuranas em nenhuma das amostras de leite analisadas. Resíduos de cloranfenicol foram detectados numa amostra de leite (leite A) com um nível de 27,2µg/g.

RESUMO

Os resíduos de 9 agentes antimicrobianos aprovados no México para uso veterinário no gado leiteiro (sulfatiazol, sulfametazine, sulfadimetoxina, sulfachloropyridazina, sulfamonometoxina, sulfametoxazol, nitrofurazona, furaltadona e furazolidona), assim como de cloranfenicol, cujo uso está proibido na produção pecuária, foram determinados no leite integral pasteurizado e comercializado na Cidade do México. Quatro indústrias de leite (A, B, C e D) foram estudadas quinzenalmente durante um ano (n=4×24=96). Os resíduos foram analisados por cromatografia de líquidos de alta pressão (HPLC) com detector UV. A porcentagem de amostras positivas a resíduos de sulfonamidas foi de 47,2% (leite A); 58,3% (leite B); 44,7% (leite C) e 50% (leite D). Os resíduos de sulfonamidas estiveram no intervalo de 1,9 a 180µg/kg. Resíduos de sulfatiazol e sulfametazina foram os mais encontrados. Somente três amostras (leite B) excederam os níveis de resíduo máximo estabelecidos para sulfonamidas. Não se detectaram resíduos de nitrofuranas em nenhuma das amostras de leite analisadas. Resíduos de cloranfenicol foram detectados numa amostra de leite (leite A) com um nível de 27,2µg/g.
tion, CV<13%) was acceptable according to the Codex Committee on Veterinary Drug Residues, which considers as acceptable the coefficients of variation below 20% (CODEX, 1989).

Results and Discussion

Residues of sulfonamides were detected in all four milk brands analyzed. In milks A, B and D, all 6 sulfonamides tested were found. However in Milk C, sulfamonemethoxine and sulfachloropyridazine were not detected (Figure 1).

The percentage of positive samples to any sulfonamide residues were: 47.2% (Milk A), 58.3% (Milk B), 44.7% (Milk C) and 50% (Milk D). These values are above those reported in milk from other countries such as Costa Rica (2%; Monge et al., 1993) and Italy (37%; Ferrini et al., 1994).

According to Honkanen and Reybroeck (1997), during the last decades the occurrence of antimicrobial residues in milk has decreased considerably in those countries where residue testing is regularly performed, and the number of inhibitor positive ex-farm milk is in general 1-5%. In Mexico there is not an antimicrobial milk residue program established which could reflect the high values of positive samples found in this study.

The sulfonamide residues with higher frequencies were sulfathiazole (33% Milk A, 20.8% Milk B, 16.7% Milk D) and sulfamerazine (37.5% Milk B, 20.8% Milk C, 16.7% Milk D).

Figure 1 presents the average residue levels for each sulfonamide tested and found in samples in the four milk brands under study. Mean values and ranges (µg/kg) were 38.2, 4.1-92.6 (Milk A); 55.4, 4.3-180 (Milk B); 21.9, 1.9-90.68 (Milk C), and 27.5, 8.7-74.4 (Milk D). The sulfonamide residues with higher mean levels were sulfamethoxazole (57.4µg/kg, Milk A, and 42.15µg/kg, Milk C), sulfamerazine (49.53µg/kg, Milk B) and sulfadiazine (37.24µg/kg, Milk B).

In Mexico no maximum residue limits (MRL) have been established for sulfonamide residues in milk. Thus, MRL values from the Codex Alimentarius, EC regulation and FDA regulations (Honkanen and Reybroeck, 1997) were considered. Only three samples (Milk B) exceeded 1.1, 1.4 and 1.8 times the MRL of 100µg/kg; the first sample presented multiple sulfonamide residues (sulfathiazole, sulfamerazine and sulfamethoxazole) with a total amount of 110.5µg/kg. The second and third ones presented sulfamerazine residues in concentrations of 141.2 and 180µg/kg, respectively.

Sulfamethazine residues, which are of high concern because of its possible carcinogenic properties (Paulson et al., 1992), were found in some of the four groups of milk samples: 8.3% (Milk A and B), 16.7% (Milk C) and 4.2% (Milk D). These values were higher than those reported in milks from Germany (2%; Grether et al., 1994) and Sweden (1.8%; Mellgren et al., 1996). The residue levels of this antimicrobial in positive samples were under the MRL established by the Codex (25µg/kg); however, if the MRL of the FDA (10µg/kg) is considered, two samples exceeded the limit, with residue levels of 18.0µg/kg (Milk A) and 23.6µg/kg (Milk C).

The residue values that violate established regulations may occur when withdrawal periods are neglected or if higher than the recommended doses are used. The presence of these unacceptable residue levels in commercial milk, which is pooled milk from different producers and, therefore, residue levels are diluted, should be taken into account. It is well known that high volumes of good quality milk can be contaminated with the milk of only one careless producer.

The high frequency of positive samples and the appearance of some cases exceeding MRL, reveals the need to establish regular residue testing programs in Mexico.

No cases of nitrofuran residues were found in milks tested. In Mexico these drugs are recommended for treatment of mastitis in dry cows (DMVNZ, 1997). Our results may reflect that these antimicrobials are used correctly only in non-lactating cows. Also it would be possible that nitrofuran residues, if present, were under the detection limits of the method used, of 4, 8 and 13µg/kg for nitrofurazone, furazolidone and furaltadone, respectively. No reports of nitrofuran residues in milk were found in the literature, probably because the use of these substances is banned in animal husbandry in several countries (Rugraff and Coulon, 1999).

Chloramphenicol residues were detected in one sample (Milk A) at levels of 27.2µg/kg. This drug is not allowed for use in dairy cattle and its presence in milk means that farmers may still use it in a non-authorized form. The presence of chloramphenicol residues in milk has also been reported in Slovenian milk (Gačnik et al., 2000), one in 217 milk samples analyzed, in levels of 4.6µg/kg.

Mexican authorities should pay more attention to the presence of antimicrobial drug residues in milk and should establish periodical residue testing programs.
this way, the occurrence of positive samples would decrease considerably.

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