

EVALUATION OF ANTIBIOTIC RESIDUE INCIDENCE IN DAIRY MILK AND CORRELATION WITH VARIOUS MILK QUALITY PARAMETERS

DETERMINAREA INCIDENȚEI REZIDUURILOR DE ANTIBIOTICE DIN LAPTELE DE VACĂ ȘI STABILIREA UNEI CORELAȚII CU ANUMIȚI INDICI DE CALITATE A LAPTELUI

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ABSTRACT | REZUMAT

In small amounts and through long-term intake, antibiotics can have undesirable effects on humans and animals. The aim of this paper was to determine the presence or absence of antibiotic residues in dairy milk, to assess the most frequently encountered antibiotic classes and to establish a correlation between positive samples and various milk quality parameters: the somatic cell count (SCC), the total number of germs (TNG) as well as protein, fat and lactose in the samples included in the study. All analyzes in this study were performed in an RENAR (Romanian Body of Accreditation) accredited laboratory. The research was carried out between May 2016 and June 2017 on a total number of 360 samples (10 samples of milk were taken each month from the three farms included in our experiments -farm A, farm B, farm C).

The main classes of identified antibiotics were: fluoroquinolones, macrolides, tetracyclines and quinolones. In 11 samples, the presence of antibiotic residues was detected above the maximum admissible limit. In positive samples, the number of somatic cells as well as the total number of germs increased. Regarding the concentration of fat and protein in the positive samples there was a decrease of the values compared to the negative samples.

Keywords: antibiotic residue, dairy milk, quality parameters

În cantități mici și prin aport îndelungat, antibioticele pot avea efecte nedorite asupra omului și animalelor. Scopul acestei lucrări a fost investigarea prezenței sau absenței reziduurilor de antibiotice, clasele de antibiotic cele mai frecvent întâlnite, precum și efectuarea unei corelații între probele depistate pozitive și anumiți parametri de calitate a laptelui: numărul de celule somatice din lapte (NCS), numărul total de germeni (NTG) și determinarea proteinei, grăsimii și lactozei. Toate analizele întreprinse în cadrul acestui studiu s-au efectuat în cadrul unui laborator acreditat RENAR (Organismul Român de Acreditare). Cercetările s-au efectuat în perioada mai 2016-iunie 2017 pe un număr de 360 de probe (s-au prelevat câte 10 probe de lapte în fiecare lună de la cele trei ferme incluse în cadrul experimentelor noastre - ferma A, ferma B, ferma C).

Principalele clase de antibiotice identificate au fost: fluoroquinolonele, macrolidele, tetraciclinele și quinolonele. La 11 probe s-a detectat prezența reziduurilor de antibiotice peste limita maximă admisă. În probele depistate pozitive s-a observat creșterea numărului de celule somatice, precum și a numărului total de germeni. Referitor la concentrația grăsimii și a proteinei din probele identificate pozitive s-a constatat scăderea valorilor față de probele negative.

Cuvinte cheie: reziduuri de antibiotice, lapte de vacă, indici de calitate

Currently, milk production, both at European and national level, is particularly high in terms of hygienic quality and less in terms of the amount of milk produced on the market (1). Raw milk must be obtained in economical units. They must possess adequate logistics in order to obtain high quality milk, which becomes a food that can be consumed in a raw state or is taken over by a specialized system, which involves collection, transportation to specific processing units (2).

The dairy and derivatives distribution market is characterized by a widespread diversification of range, where quality management becomes the key to success for both producers and processors. For all, quality is the ultimate determinant of profit. We are in front of a large offer of dairy products but, as consumers, we cannot always trust in the processing methods used by the producers (5).

A strictly objective analysis shows that none of the foods considered to be basic is an ideal food (not even milk, as it may have adverse effects on consumers), all the more so as the product composition can be affected by changes in the housing system, their welfare

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and health, and last but not least by the way it is processed for consumption (5). Consequently, effective knowledge and control of composition and hygienic integrity indicators of milk will influence its quality and that of milk products destined for consumption (6). The presence of antibiotics in milk is undesirable from the point of view of milk innocuity and control of pasteurization efficiency. In small quantities and through long-term intake, antibiotics can also have undesirable effects on humans and animals. The undesirable effects to the health of consumers may be direct and manifest in the form of allergic or even toxic reactions. Indirect effects are also important due to creation of antibiotic-resistant micro-organisms. Furthermore, samples containing antibiotic residues are accompanied by increases in somatic cell population and total germ counts (7).

The purpose of this paper was to determine the presence or absence of antibiotic residues in dairy milk, to assess the most frequently encountered antibiotic classes, and to establish a correlation between positive samples and various milk quality parameters: the somatic cell count (SCC), the total number of germs (TNG) as well as protein, fat and lactose in the samples included in the study.

MATERIALS AND METHODS

The research was carried out between May 2016 and June 2017 on a number of 360 samples (10 samples of milk were taken each month from the three farms included in our experiments - farm A, farm B, farm C). The sampling and preparation of the samples took into account several important aspects: proper homogenization of milk prior to collection, sufficient volume to perform for analyzes and repeatability of some techniques, keeping the samples at a suitable temperature (0-4 °C), as well as transporting them as soon as possible to the laboratory. All analyzes performed in this study were performed in an RENAR accredited laboratory. For the detection of antibiotic residues in milk, the LC-MS/MS (Tandem Mass Spectrometry coupled with liquid chromatography) technique was used.

RESULTS AND DISCUSSIONS

The main classes of antibiotics identified in farm A were fluoroquinolones (25% of samples), macrolides (21.66% of samples), tetracyclines (in 19.58% of samples), sulfonamides (6.66% of samples) and qui-

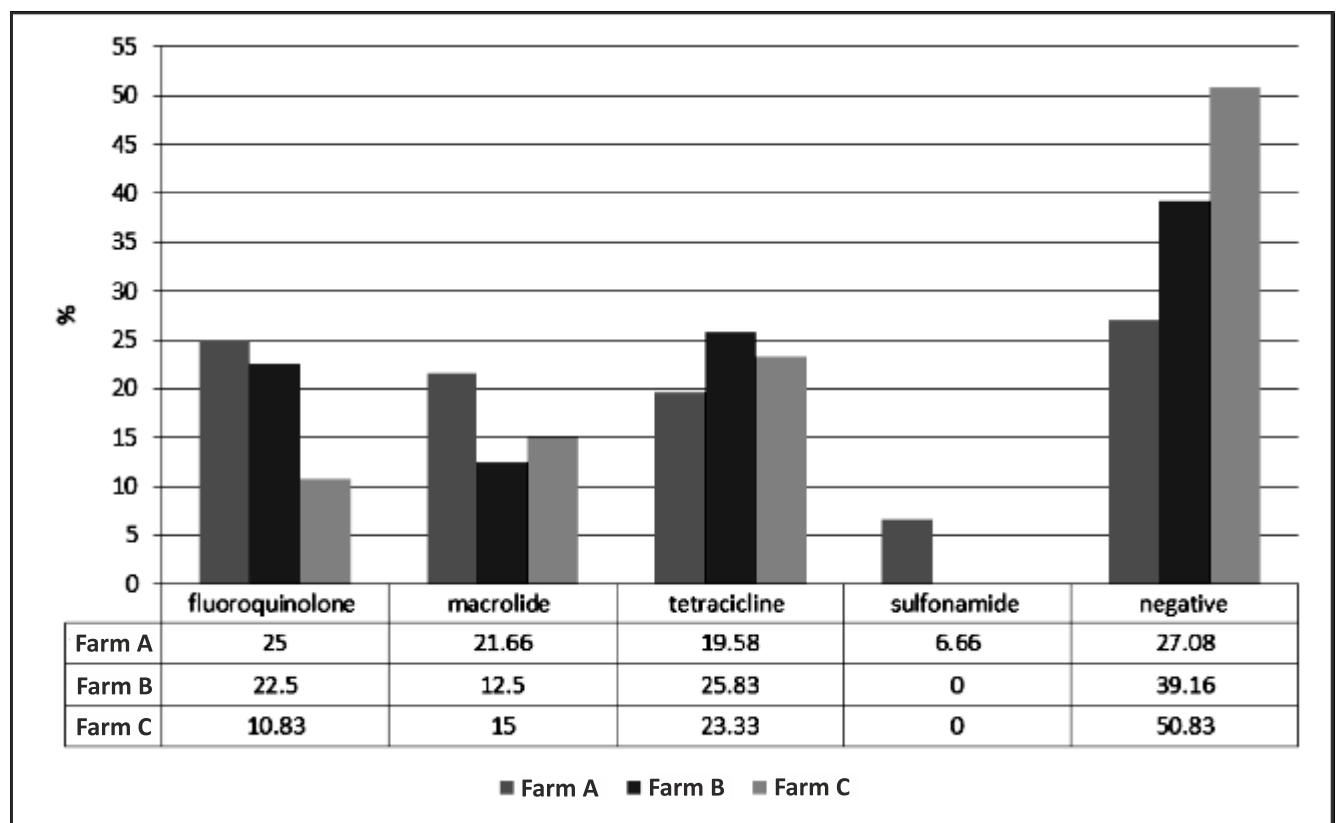


Fig. 1. Main classes of identified antibiotics

Table 2.

Concentration of macrolide residues determined from milk samples

		Average ($\mu\text{g/l}$)	Minimum ($\mu\text{g/l}$)	Maximum ($\mu\text{g/l}$)	DS ($\mu\text{g/l}$)	LMR ($\mu\text{g/kg}$)
Erythromycin	Farm A	17,24	9,70	24,60	$\pm 2,55$	40
	Farm B	14,24	8,50	19,22	$\pm 2,55$	
	Farm C	7,24	1,70	12,78	$\pm 2,55$	
Spiramycin	Farm A	12,23	7,70	14,80	$\pm 2,72$	200
	Farm B	77,23	7,70	140,80	$\pm 2,72$	
	Farm C	22,23	4,70	48,80	$\pm 2,72$	
Tilmicosin	Farm A	11,70	8,70	13,40	$\pm 1,03$	50
	Farm B	8,70	4,70	12,40	$\pm 1,03$	
Tylosin	Farm A	6,54	5,30	6,80	$\pm 2,18$	50
Legend: DS - standard deviation; MRL - maximum limit of residues						

Table 3.

Concentration of tetracycline residues determined from milk samples

		Average (µg/l)	Minimum (µg/l)	Maximum (µg/l)	DS (µg/l)	LMR (µg/kg)
Oxitetraciclina+4 EpiOxytetracycline	Farm A	90,25	59,70	110,20	±2,74	100
	Farm B	45,25	4,70	109,70	±7,74	
	Farm C	14,25	0,70	45,20	±5,24	
Tetracycline+4 EpiTetracycline	Farm A	87,20	67,70	104,80	±1,72	100
	Farm B	27,20	11,70	45,27	±3,72	
	Farm C	12,20	1,05	28,80	±8,72	
Chlortetracycline+4 EpiChlortetracycline	Farm A	65,70	58,70	69,40	±0,54	100
	Farm B	25,70	18,70	49,40	±10,5	
Legend: DS - standard deviation; MRL - maximum limit of residues						

Table 4.

Values of milk quality indicators in analyzed samples

Investigated parameters	Negative samples	Positive samples	Normal values
NCS (x 1000/ml)	121.47 $\pm$ 21.2	119.41 $\pm$ 65.7	≤ 400.000
NTG (x 1000/ml)	44.11 $\pm$ 7.6	347.6 $\pm$ 27.5	≤ 100.000
Fat (%)	4.17 $\pm$ 0.14	3.1 $\pm$ 0.45	3.5-4.5
Protein (%)	3.27 $\pm$ 0.24	2.98 $\pm$ 0.78	3-3.5

and 3.27% for protein. The mean somatic cell count was 121,470 (x 1000)/ml, and the total germ count was 44.11 ± 9.6 (x 1000)/ml.

In milk samples containing antibiotic residues above the maximum admissible limit, an increase of the total number of germs - 347.6 (x1000) / ml and the number of somatic cells - 121.44 (x1000)/ml was found. At the same time, the percentage of fat (3.1 ± 0.45) and protein (2.98 ± 0.78) was decreased. In a study on the evaluation of the incidence of antibiotic residues in milk and their influence on quality raw milk, Grădinaru et al., 2009, identified as positive, (i.e. the presence of antibiotics in milk), a total number of 21 from 372 samples. The same study showed close values of the main milk quality indicators as compared to those expressed in our investigations.

CONCLUSIONS

The main classes of antibiotics that were identified are: fluoroquinolones, macrolides, tetracyclines and quinolones. In 11 samples, the presence of antibiotic residues was detected over the maximum admissible limit.

An increase of the number of somatic and total number of germs was observed in positive samples.

The concentration of fat and protein decreased in the positively identified samples as compared to negative samples.

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