

Changes of Iodine Content in Milk of Cows treated with Betadine

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Abstract

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The purpose of this study was to assess the changes of iodine content in milk associated with the treatment of milch cows with the disinfecting preparation Betadine on basis of active iodine. Altogether 17 samples were taken from milch cows treated after milking by immersion of teats into Betadine solution; further 3 samples from cows after uterine irrigation. Ten check samples from milch cows without treatment were analyzed too. The content of iodine in milk was determined by the sensitive spectrophotometric method based on the Sandell-Kolthoff reaction brought to an end with addition of ammonium ferrous sulphate. Organic matter of milk was destroyed by wet acid mineralization. Average concentration and confidence interval of iodine content in check samples were $58 \pm 15 \mu\text{g/l}$. Milk from cows treated after milking with Betadine was not contaminated with iodine ($53 \pm 18 \mu\text{g/l}$), increased variable concentration of iodine (from 283 to 1078 $\mu\text{g/l}$) was found in the milk from cows after uterine irrigation by Betadine.

Key words: spectrophotometry; milk; iodine; disinfectant; Betadine; Sandell-Kolthoff reaction

Content of iodine in milk generally depends on regional geological factors, on iodine intake by feed including nutritional supplements and also on the use of iodophors as disinfectants, therefore it lies in the wide range from 10 $\mu\text{g/l}$ to 800 $\mu\text{g/l}$, average content is indicated about 260 $\mu\text{g/l}$ (FLYNN & POWER 1985). Lower value (179.5 $\mu\text{g/l}$) is presented in the Czech Republic (BORKOVCOVÁ *et al.* 1999). In NOLAN and PURNELL (1977) opinion up to 100 μg iodine per litre can occur naturally in cow milk and it is very rare for this level to be exceeded in milk obtained directly from the cow.

Recently there has been a marked increase in the use of iodophors in the dairy industry because they have an excellent disinfecting effect and low toxicity. The wide use of iodophors however, and the possible subsequent high levels of iodine residues due to bad dairy practice could give rise to a risk of iodine-induced thyrotoxicosis in susceptible members of the population or difficulties in dairy technology (NOLAN & PURNELL 1977). Disinfecting properties of iodophors including Betadine were studied by HARTMANOVÁ (1996). Iodine is bound in Betadine to the macromolecular carrier – polyvinylpyrrolidone (PVP).

Various methods have been published for the determination of total iodine in milk. Preparation of samples is usually based on digestion procedure. Wet ashing was used e.g., by NOLAN and PURNELL (1977) or WIECHEN and KOCK (1984), dry ashing was utilized by TUŠL (1976) or

AYIANNIDIS and VOULGAROPOULOS (1988). The most commonly used assays are variations of a colorimetric procedure based on the catalytic effect of iodide in the redox reaction $2\text{Ce}^{4+} + \text{As}^{3+} \rightarrow 2\text{Ce}^{3+} + \text{As}^{5+}$ (SANDELL & KOLTHOFF 1934). In the absence of iodide this reaction proceeds with extreme slowness, traces of iodide increase the speed of the reaction enormously. Course of a reaction is monitored directly by measuring the absorbance at 380 nm (due to Ce^{4+} that decreases with time) or indirectly after terminating of the reaction. It can be brought to an end with addition of ammonium ferrous sulphate (TUŠL 1976), created ferric ions are assessed as a complex with thiocyanate spectrophotometrically at 525 nm. Another possibility is the use of brucine, colour product is measured at 430 nm (BARKLEY & THOMPSON 1960).

MATERIAL AND METHODS

Milk Samples: Altogether thirty samples from the milk farm Taurus (Rtyně v Podkrkonoší, Trutnov district) were examined. Samples numbered 1 to 17 (Table 1) were taken from milch cows treated after milking by immersion of teats into Betadine solution (dilution with water 1 : 10, always newly prepared) for the 10 preceding days. The mammary gland was washed with water and dried before milking. Samples numbered 18 to 20 were taken from cows after uterine irrigation also with Betadine solution (1 : 10)

24 hours before sampling. Ten check samples (No 21-30) were taken from milch cows without treatment.

Reagents: Disinfectant Betadine liq. (10 mg of active iodine per ml) was supplied by Egis Pharmaceuticals Ltd (Budapest, Hungary). The reagents and water for iodine assay must be as free as possible of iodine. The glassware was decontaminated according to a procedure described by WIECHEN and KOCK (1984). The acids (H_2SO_4 , $HClO_4$, HNO_3) were obtained from Analytica (Prague, CR), ceric ammonium sulphate and arsenic trioxide were purchased from Merck (Darmstadt, Germany), other chemicals were from Lachema (Brno, CR). All solutions were prepared using deionized water. All reagents were A.R. quality or better.

Apparatus: Mineralization of samples was performed in Digestion system 20, 1015 Digester with Autostep 1012 Controller (Tecator, Höganäs, Sweden). Digestion unit was equipped with aluminium inserts for small test tubes. Absorbance was measured by UV-VIS spectrophotometer Carry 50 (Varian, Mulgrave, Australia).

Iodine Determination: Samples (2 ml of milk) were digested in tubes by a mixture of acids (9 ml) (WIECHEN & KOCK 1984). The tubes were placed into the combustion unit. The combustion process proceeds for 7.5 hours with a regulated temperature increase up to 200°C. Further treatment of samples and preparation of calibration samples carried out by a procedure described by WIECHEN and KOCK (1984) including dilution of mineralizate to 25 ml. As the calibration and the evaluation of the results are considerably influenced by the blind test, three blind tests are prepared and their average is used after possible elimination of an extreme value (MELOUN & MILITKÝ 1998).

Two ml of diluted mineralizate and calibration samples are taken for further analysis, which was performed according to a modified procedure described by TUŠL (1976).

Potassium iodate was used instead of potassium iodide for calibration purposes. In the first step of determination of iodine, the iodate ions, formed in the digestion mixture, are reduced by 4 ml of the working solution of As^{3+} (including the calibrating series). After 5 min, 0.5 ml of the ceric salt solution must be added into all test tubes within 30 s. The tubes as a whole are placed in water bath having the temperature of 50°C. The test tubes are removed from the bath after precisely 50 min and dipped in cold water. Heating time and temperature were optimised for gain sensitivity. Addition of 0.5 ml of the ammonium ferrous sulphate solution stops the reaction. After the solution gets discoloured, addition of 0.5 ml of thiocyanate solution induces red coloration. Its absorbance is measured after 5 min using a spectrophotometer at 525 nm relative to water.

There are two possibilities to evaluate calibration dependence. Iodine concentration in the reaction mixture can be plotted vs. absorbance (TUŠL 1976) or the difference between logarithms of absorbance in blind sample and those in calibration solution (WIECHEN & KOCK 1984). The later relationship provided a more favourable correlation coefficient after fitting a straight line; therefore, it was used for the evaluation of results. An example of calibration graph is shown in Fig. 1, where iodine concentration is the amount of iodine per ml of reaction mixture.

Two mineralizations were performed for iodine determination by at least two analyses for each mineralizate. Extreme values were eliminated using the Horn procedure (MELOUN & MILITKÝ 1998).

RESULTS AND DISCUSSION

The obtained results of the analyses are summarised in Table 1 and 2. The average values for check samples and samples numbered 1-17 are in agreement with KURSA *et*

Table 1. Iodine content [$\mu g/l$] in milk of cows treated with Betadine

| Sample | Iodine content | SD | n | Sample | Iodine content | SD | n | Sample | Iodine content | SD | n |
|--------|----------------|----|---|--------|----------------|----|---|--------|----------------|----|---|
| 1 | 53 | 7 | 5 | 11 | 45 | 10 | 4 | 21 | 42 | 7 | 4 |
| 2 | 38 | 6 | 5 | 12 | 72 | 2 | 4 | 22 | 23 | 3 | 4 |
| 3 | 27 | 5 | 4 | 13 | 123 | 14 | 4 | 23 | 80 | 6 | 5 |
| 4 | 117 | 8 | 4 | 14 | 41 | 1 | 4 | 24 | 78 | 7 | 5 |
| 5 | 64 | 10 | 5 | 15 | 31 | 5 | 4 | 25 | 45 | 11 | 4 |
| 6 | 38 | 8 | 5 | 16 | 23 | 2 | 5 | 26 | 31 | 3 | 5 |
| 7 | 39 | 2 | 4 | 17 | 50 | 9 | 5 | 27 | 34 | 2 | 4 |
| 8 | 52 | 5 | 4 | 18 | 1078 | 81 | 4 | 28 | 75 | 7 | 5 |
| 9 | 31 | 6 | 5 | 19 | 307 | 44 | 4 | 29 | 69 | 7 | 5 |
| 10 | 59 | 12 | 5 | 20 | 283 | 25 | 4 | 30 | 100 | 12 | 5 |

Samples 1-17 mammary gland treated; samples 18-20 uterine irrigation performed; 21-30 check samples; SD - standard deviation, n - number of iodine determinations taken for further evaluation

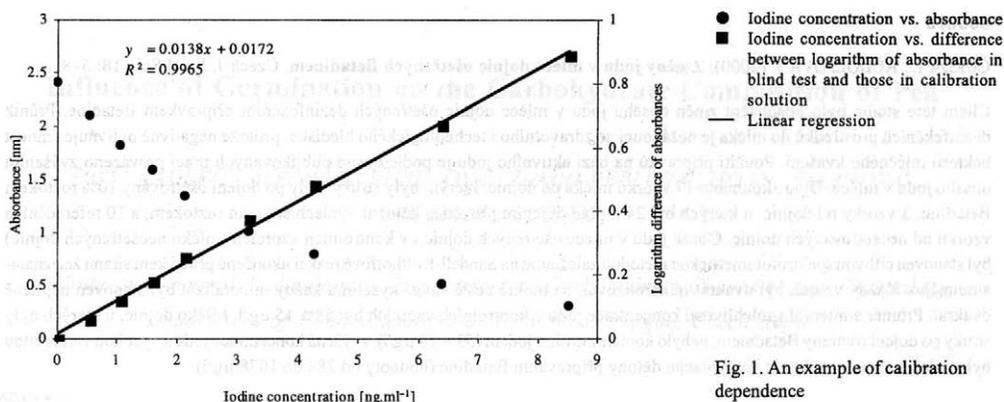


Fig. 1. An example of calibration dependence

Table 2. Survey of iodine content [$\mu\text{g/l}$] in milk samples

| Samples | Content | | | SD |
|---------|---------|---------|---------|----|
| | minimal | maximal | average | |
| 1–17 | 23 | 123 | 53 | 28 |
| 18–20 | 283 | 1078 | – | – |
| 21–30 | 23 | 100 | 58 | 26 |

For legend see Table 1

al. (1998), who presents that more than 80% of milk produced in the Czech Republic do not contain even 100 μg of iodine per litre. It can be considered rather low if compared with iodine concentration in milk found by BORKOVCOVÁ *et al.* (1999). The difference between the iodine content in the check samples and the iodine content in the samples after treatment of teats is not statistically significant by Student's *t*-test (MELOUN & MILITKÝ 1998). FLYNN and POWER (1985) review that the improper use of iodophors for teat disinfection increases the iodine content in cow's milk by 120 to 700 $\mu\text{g/l}$ and iodine contamination of milk occurs as a result of absorption through the skin rather than by surface contamination of the teat. Properties of macromolecular carrier in Betadine (PVP) could particularly influence extent of absorption through the skin and consecutively the low concentration of iodine in milk. Samples No 18 through 20 display a well-documented increase in iodine levels (Table 1). These concentrations are approx. up to twenty times higher than the average of the check samples, therefore it would be interesting to observe time dependence of iodine content after uterine irrigation.

We can conclude that correct treatment of teats with Betadine (i.e., proper dilution of Betadine, short time of teats dipping) does not increase the content of iodine in milk. High iodine concentration was found in milk after uterine irrigation.

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Souhrn

ČURDA L., RUDOLFOVÁ J. (2000): **Změny jodu v mléce dojnic ošetřených Betadinem**. Czech J. Food Sci., 18: 5–8.

Cílem této studie bylo posouzení změn obsahu jodu v mléce dojnic ošetřených dezinfekčním přípravkem Betadine. Průnik dezinfekčních prostředků do mléka je nežádoucí ze zdravotního i technologického hlediska, protože negativně ovlivňuje činnost bakterií mléčného kvašení. Použití přípravků na bázi aktivního jodu je podle dosud publikovaných prací prováděno zvýšením obsahu jodu v mléce. Bylo zkoumáno 17 vzorků mléka od dojnic, kterým byly struky vždy po dojení ošetřovány 10% roztokem Betadinu, 3 vzorky od dojnic, u kterých byl 24 h před dojením proveden děložní výplach stejným roztokem, a 10 referenčních vzorků od neošetřovaných dojnic. Obsah jodu v mléce ošetřených dojnic i v kontrolních vzorcích (mléko neošetřených dojnic) byl stanoven citlivou spektrofotometrickou metodou založenou na Sandell-Kolthoffově reakci ukončené přidávkem siranu železato-amonného. Každý vzorek byl dvakrát mineralizován na mokré cestě směsí kyselin a každý mineralizát byl stanoven nejméně dvakrát. Průměr a interval spolehlivosti koncentrace jodu v kontrolních vzorcích byl $58 \pm 15 \mu\text{g/l}$. Mléko dojnic, u kterých byly struky po dojení ošetřeny Betadinem, nebylo kontaminováno jodem ($53 \pm 18 \mu\text{g/l}$), zvýšená koncentrace jodu s vysokou variabilitou byla nalezena v mléce dojnic po výplachu dělohy přípravkem Betadine (hodnoty od 283 do 1078 $\mu\text{g/l}$).

Klíčová slova: spektrofotometrie; mléko; jod; dezinfekční prostředky; Betadine; Sandell-Kolthoffova reakce

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