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Original Research Article

MONITORING OF MARKETED PORK OF ASSAM AND MEGHALAYA FOR CIPROFLOXACIN RESIDUES

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Abstract: The study was undertaken to monitor marketed pork of Assam and its neighboring state Meghalaya for Ciprofloxacin residues using Ultra High Performance Liquid Chromatography (UHPLC). 300 samples of marketed pork of Assam and Meghalaya were collected for the study. The samples after collection were preserved at -20°C. Recoveries of ciprofloxacin in pork ranged from 88-98%. Out of the tested samples, 18 samples were detected to be positive for trace residues of Ciprofloxacin which were well below the Maximum Residue Limit (MRL) value.

Keywords: Ciprofloxacin, Monitoring, Pork, Residues, UHPLC

Introduction: Fluoroquinolones are synthetic broad-spectrum antibiotics. These antimicrobial substances are used for preventive and therapeutic purposes in farm animals, especially in pigs (Salehzadeh *et al*., 2007). These agents have a wide range of antibacterial activity and so have been in use in veterinary medicine. They are mainly used against urinary, pulmonary and digestive infections (Bauditz, 1990). In fact they have been used successfully to treat infection caused by Gram positive and Gram negative bacteria. These compounds act by inhibiting the bacterial DNA-gyrase (Gilles *et al.*, 1991). Chemical structure of fluroquinolones when changed, an increase in the spectrum of activity and potency of the drugs is seen (Spoo *et al*., 1995).

Ciprofloxacin was first introduced into human medicine and is also in use in veterinary medicine (Nouws *et al*., 1988). Ciprofloxacin residues are reported in animal tissues (Schneider, 2001; Cornejo *et al*., 2011). The indiscriminate use of this agent possesses a considerable risk due to the presence of residues in pork meant for human consumption. Ciprofloxacin may be found as residues in meat of slaughtered pig. The FAO /WHO have recommended Maximum Residue Limits

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(MRL) for Ciprofloxacin in animal tissues (FAO/WHO, 2002).

The North-Eastern States of India is characterized by a high proportion of tribal people for whom pig rearing is integral to their way of life and pig meat is considered as an important food item. Thus, the present study was undertaken for monitoring of Ciprofloxacin residues in pork samples from various areas of Assam and its neighboring state Meghalaya using UHPLC.

Materials and Methods: 300 samples of swine tissues comprising of Muscle, Kidney and Liver were collected from different markets of Assam and Meghalaya (Table 1). Representative samples weighing 30 g each belonging to same carcass were wrapped in polythene bags and transported in thermo-cooled containers jacketed with ice. The samples were stored at -20ºC till the time of processing. The levels of Ciprofloxacin were determined using a UHPLC system of Make: Dionex with Diode Array Detector (DAD) operated at 277 nm. The samples were separated on a RP-C_{18} column and were eluted with a mobile phase of mixture of Water and Acetonitrile in the ratio of 67:33 v/v. The isocratic mode was run at a flow rate of 1 ml/min.

Chemical and reagents: Ciprofloxacin standard (Dr. Ehrenstofer, Germany); HPLC grade Acetonitrile (Fisher), Methanol (Fisher); other chemicals and solvents of analytical grade and HPLC grade water (Fisher) were used for the study.

Preparation of sample: The fascia and fat of pork were removed and then cut into small pieces. 10 g of the sample was taken in a blender and to it added equal volume of distilled water. Ten grams of each blended sample was transferred to centrifuge tube. After few minutes 10 ml of acetonitrile was added. The sample was ultrasonicated and left undisturbed for 10 min. The samples were centrifuged and the collected supernatant was filtered. The filtrate then was passed through C_{18} polymeric cartridge after which it was further filtered using 0.22µm filter paper.

Results and Discussion: Linear calibration curve of Ciprofloxacin having correlation coefficient (R^2) of 0.99 was achieved. Recoveries of ciprofloxacin in pork ranged from 88-98%. This was similar to that reported by Cinquina *et al*.,2003. Ciprofloxacin was eluted under isocratic conditions. The excitation/emission wavelengths used for the UV detection were 277 nm for Ciprofloxacin. The separation of the analytes was achieved in less than 5 mins. Acetonitrile was effective in the deproteinization of pork samples and in the isolation of analytes from spiked samples. This method allows the determination of residues of ciprofloxacin in different matrices with higher sensitivity.

Over all, nine zones within the state of Assam and five zones from the state of Meghalaya have been selected and a total of 300 samples of pork comprising of muscle, liver and kidney were collected. A total of 18 pork samples were detected to be positive of Ciprofloxacin residue. As listed in Table 2, only 4 kidney, 6 liver and 8 muscle samples showed detectable ciprofloxacin residues using UHPLC. All the samples were below the permissible limit. Residue level of ciprofloxacin detected in muscle, kidney and liver were 0.009-0.018 $\mu g.g^{-1}$, 0.016-0.025 $\mu g.g^{-1}$ and 0.010-0.023 $\mu g.g^{-1}$ respectively.

A total of 11(eleven) samples from Assam and 7(seven) samples of pork from the state of Meghalaya were detected to be positive of residues of Ciprofloxacin as given in Table 3. From the state of Assam, Guwahati recorded the maximum number of positive samples while from the state of Meghalaya, maximum numbers of positive samples were detected from shillong.

Conclusion: 300 samples of pork were collected from different pork markets of Assam and Meghalaya. Out of the screened samples, 18 samples were detected to be positive for trace

residues of Ciprofloxacin using UHPLC which were all below the MRL.

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Table 1: Collection of Pork Samples

PLACE	KIDNEY	LIVER	MUSCLE	TOTAL
ASSAM				
Guwahati	18	18	18	**54**
Morigaon	7	7	7	**21**
Nagaon	8	8	8	**24**
Mangaldai	5	5	5	**15**
Dhemaji	7	7	7	**21**
Karbi Anglong	6	6	6	**18**
Jorhat	5	5	5	**15**
Kokrajhar	7	7	7	**21**
Dibrugarh	7	7	7	**21**
MEGHALAYA				
Shillong	6	6	6	**18**
Tura	7	7	7	**21**
Barapani	5	5	5	**15**
Burnihat	5	5	5	**15**
9th Mile	7	7	7	**21**
TOTAL	**100**	**100**	**100**	**300**

Table 2: Ciprofloxacin Residue Level in Swine Tissues

Tissue	No. of positive samples detected	Level of residue detected (µg /g)
Kidney	4	0.016-0.025
Liver	6	0.010-0.023
Muscle	8	0.009-0.018
TOTAL	**18**	**----**

Table 3: Locationwise Distribution of Ciprofloxacin Residues in Pork Samples

PLACE	TISSUES DETECTED WITH RESIDUES			TOTAL
	KIDNEY	LIVER	MUSCLE	
ASSAM				
Guwahati	1	2	3	**6**
Morigaon	ND	ND	ND	**ND**
Nagaon	ND	1	ND	**1**
Mangaldai	ND	ND	ND	**ND**
Dhemaji	ND	ND	1	**1**
Karbi Anglong	1	ND	ND	**1**
Jorhat	ND	ND	1	**1**
Kokrajhar	ND	1	ND	**1**
Dibrugarh	ND	ND	ND	**ND**
TOTAL(ASSAM)	**2**	**4**	**5**	**11**
MEGHALAYA				
Shillong	1	2	1	**4**
Tura	ND	ND	1	**1**
Barapani	ND	ND	ND	**ND**
Burnihat	ND	ND	1	**1**
9th Mile	1	ND	ND	**1**
TOTAL(MEGHALAYA)	**2**	**2**	**3**	**7**
GRAND TOTAL	**4**	**6**	**8**	

ND-NOT DETECTED