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Does your milling procedure for cereals influence your pesticide residue results?

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Introduction: Previous studies at the EURL-CF has shown that there was a relation between decreasing particle size and increased extraction efficiency of incurred pesticide residues in cereals. To get an overview of how EU laboratories perform milling of cereal grains, a survey among 23 NRL-CFs was performed, (Table 1).

Procedure: Excess of grains from production of EUPT-C3 (oat) and EUPT-C4 (rye) Test Items with incurred pesticides was used as test materials (Table 2). Each participating NRL milled 100 g of grain, both oat and rye, with their routine mill and procedure. At the EURL-CF, half of each sample was separated into fractions, using a vibratory sieve shaker, to determine the particle size distribution (Picture 1 and 2). Residue levels of the 23 different pesticides were determined by QuEChERS.

Particle size distribution: Large variation was seen in particle size. The particle size distributions showed that samples milled by knife and hammer mill were very un-homogenous and consisted of a large amount of particles larger than 0.7 mm. Samples milled by universal horizontal and centrifugal mill (at 0.5 or 1.0 mm) were more homogenous and consisted primarily of particles smaller than 0.7 mm. The distributions of the samples with the smallest and the largest particles for oat and rye are presented in Figure 1. The rye and oat samples with the smallest particles were milled frozen by centrifugal mill with sieve mesh size at 0.5 mm

The relative particle size, RPS of each sample was calculated as:

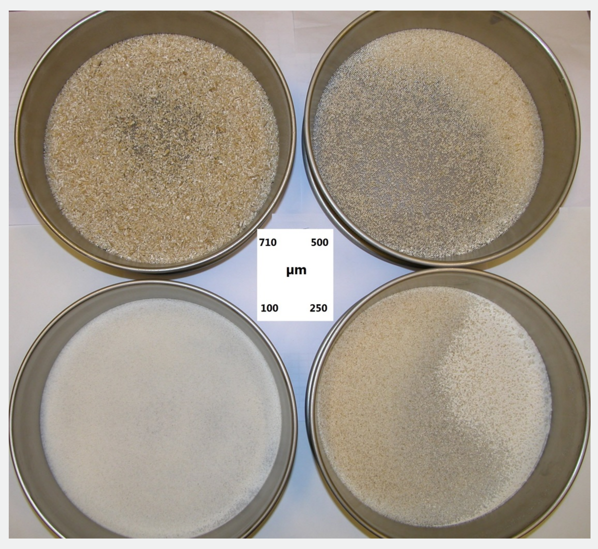
$$\sum_{i=1}^{n} \text{Average between sieve n and sieve n−1 in µm} \ast \text{Sample Amount in sieve n (g)}$$

The RPS ranged from 16-155 for rye and 24-129 for oat (Figure 2). As seen, in Figure 2 it is also possible to obtain primarily small particle size with knife mill. For knife mills the results are depended on the milling time, quality of the knife etc.

Pesticide residues: Results for oat showed that particle size had a clear influence on the extraction efficiency (Figure 3). The recoveries obtained for the samples with the smallest particles was up to 40% higher than the recoveries for the largest particles. For rye the influence was not so clear. However, also here significant differences were seen in samples with low and high RPS (Figure 3). We expect the difference between oat and rye to be that the pesticides are more evenly distributed in the oat kernel because it is protected against direct exposure of pesticides by the hulls.

Conclusion: Milling of cereal grains at the NRLs resulted in very different particle size distributions. Laboratories using centrifugal mill with sieve size of 0.5 or 1 mm, resulted in more homogenous and primarily of smaller particles. Thus, pesticide residue results for cereals depended on the degree of milling and higher results were achieved for samples with small RPS.

Picture 1 Vibratory sieve shaker



Picture 2 Sieves with samples

Table 1. Laboratories that contributed to the study

Laboratory	City	Country
Central Laboratory for Chemical Testing and Control	Sofia	Bulgaria
State General Laboratory	Nicosia	Cyprus
Czech Agriculture and Food Inspection Authority	Praha	Czech Republic
Central Institute for Supervising and Testing in Agriculture	Brno	Czech Republic
Danish Veterinary and Food Administration	Ringsted	Denmark
Agricultural Research Centre	Saku	Estonia
Finnish Customs Laboratory	Espoo	Finland
Service Commun des Laboratoires	Massy Cedex	France
Federal Office of Consumer Protec and Food Safety	Berlin	Germany
Benaki Phytopathological Institute	Kifissia	Greece
National Food Chain Safety Office	Velence	Hungary
National Food Chain Safety Office	*Miskolc*	*Hungary*
Pesticide Control Laboratory, Dep. of Agriculture, Fisheries and Food	Co. Kildare	Ireland
Centro di referenza nazionale	Genova	Italy
Institute of Food Safety (BIOR)	Riga	Latvia
Institute of Plant Protection,	Poznan	Poland
INIAV- Pesticide Residues Laboratory	Oeiras	Portugal
State Veterinary and Food Institute	Bratislava	Slovakia
National Laboratory of Health, Environment and Food	Maribor	Slovenia
National Centre for Food	Majadahonda	Spain
Laboratorio Arbitral Agroalimentario	Madrid	Spain
National Food Agency, Science Department	Uppsala	Sweden
Fera Science Ltd	York	United Kingdom

Table 2 Incurred pesticide residue levels in the test materials in mg/kg

Incurred pesticides	Oat	Rye
Azoxystrobin	0.184	0.31
Carbendazim	0.44	1.12
Chlorpyrifos	1.054	
Chlorpyrifos-methyl		0.094
Cyproconazole	1.013	
Cyprodinil	0.075	
Deltamethrin		0.057
Fenbuconazole	0.554	-
Fenpropimorph	0.162	2.06
Fenvalerate	0.099	
Fludioxonil	0.108	
Fluquinconazole		0.629
Flusilazole	0.814	
Flutriafol		2.81
Kresoxim-methyl		0.425
Lambda-cyhalothrin	0.033	0.036
Malathion	0.011	0.094
Metconazole	0.564	
Pirimiphos-methyl		0.048
Pyraclostrobin	0.816	
Spiroxamin		1.15
Tebuconazole	1.787	
Triadimenol		1.83

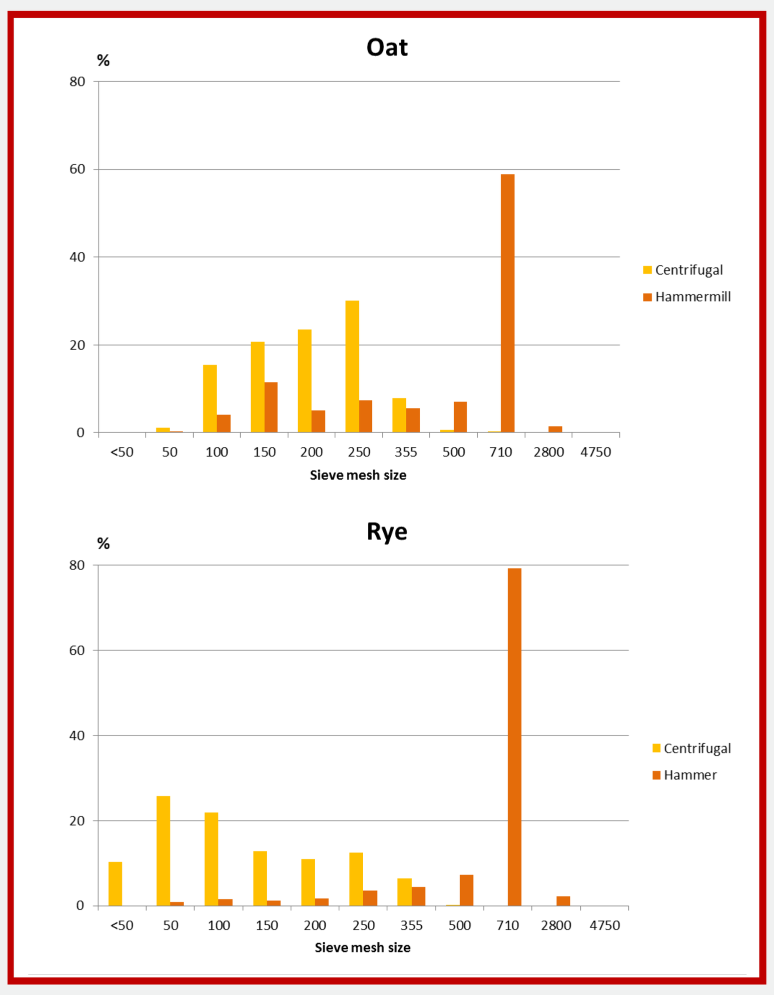


Figure 1 The particle size distributions of the two samples with the smallest and the largest particles for both oat and rye.

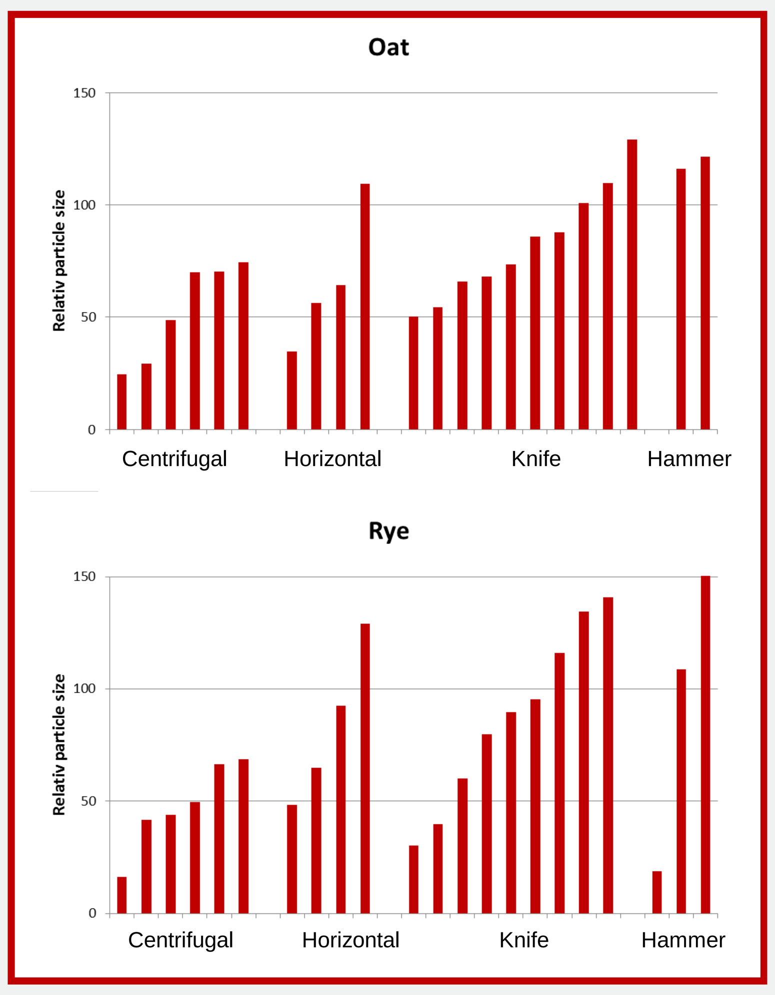


Figure 2 The relative particle size, RPS for all samples of oat and rye grouped by type of mill.

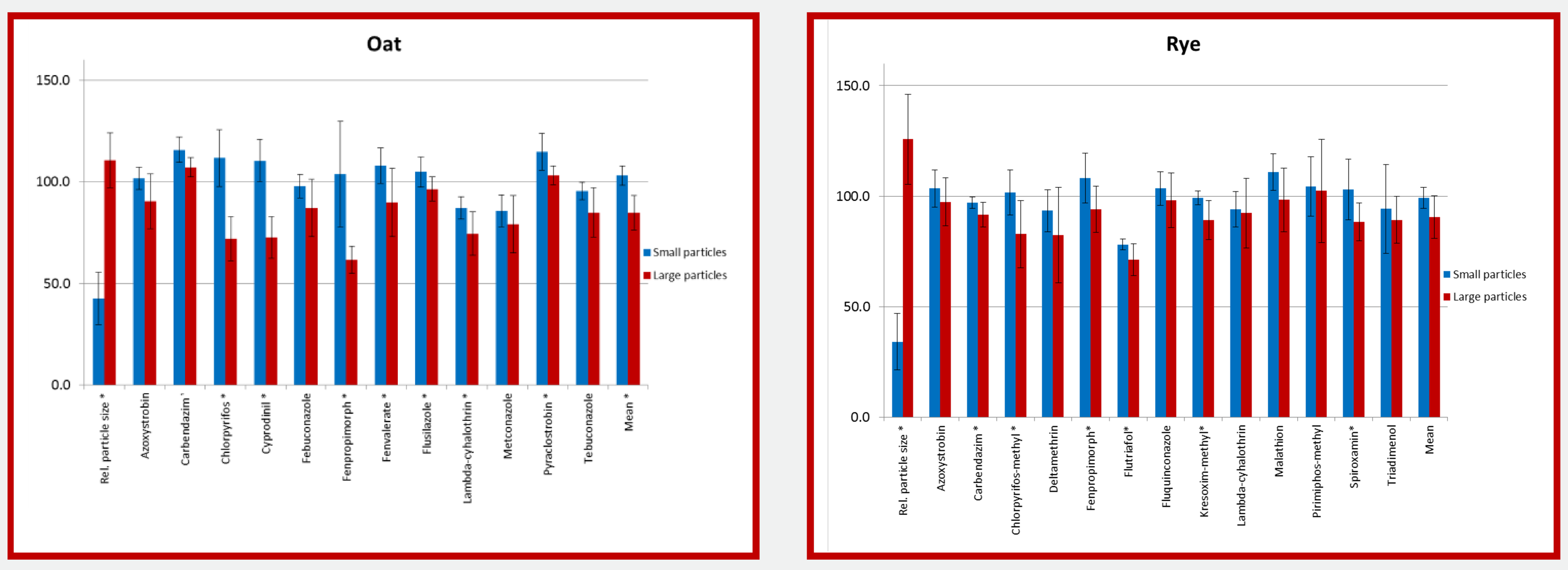


Figure 3 Relative particle Size, RPS and pesticide recoveries for the seven samples with the smallest and largest RPS. Differences are significant (t-test, P<0.05) for pesticides marked with *.

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