Evaluating the Norwegian standard methods for the total and extractable heavy metal concentration in compost

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CODIS 2008
February 27-29
Solothurn, Switzerland

Background

- **Herbage concentration of Zn and Cu is low in organic farming**
- **There is a need for organic fertilisers with high concentration of Zn and Cu in organic farming, but regulations do not allow the use of these organic fertilisers in organic farming**
- **You are allowed to use inorganic Zn or Cu in organic farming when plant deficiency is detected**
- **Objective**

  Are today's methods for determination of plant available Cu and Zn and total concentration of Cu, Zn, Ni, Cr, Cd and Pb "useful" and can they be replaced by other methods

Compost quality - regulations

- **Hygienic and stabilized**
- **Concentration of organic pollutants as low as possible**
- **Content of unwanted items should be as low as possible**

- **Concentration of heavy metals according to a list**

mg/kg DM	Organic	0	I	II	III	Max conc. in agric. soil when using class I or II
Cadmium	0.7	0.4	0.8	2	5	1
Lead	45	40	60	80	200	50
Mercury	0.4	0.2	0.6	3	5	1
Nickel	25	20	30	50	80	30
Zinc	200	150	400	800	1500	150
Copper	70	50	150	650	1000	50
Chromium	70	50	60	100	150	100

Method - plant uptake

- **Greenhouse experiment**

  Ryegrass (*Lolium perenne*) was established and cut twice before the experiment started

  5 g composts (~50 ton/ha) supplied at 5 cm depth, the sand soil beneath was replaced

  Fertilised at growth day 1, 37 and 67

  Harvested at 2 cm height at growth day 15, 30, 45, 60 and 75

  Samples dried and grinded prior to determination of Zn, Cu, Cd, Ni, Cr and Pb

Method – total concentration

- **Wet digestion**

  Microwave HNO_3 digestion, ultraclave at 240 ºC

  ICP/MS

- **XRF**

  X-ray Fluorescence analyser (XRF) directly on grinded (1mm) samples in plastic bags

Method - extractable

- **CAT method**
  - Analysed at Jordforsk Lab, a part of Eurofins (October 2007)
  - 0.01M CaCl2 + 0.002M DTPA, pH 2.6-2.65
  - ICP-MS
- **Water extractable**
  - 4 g compost shaken in 40 ml MQ water for 24 h, 20 ºC
  - Ultracentrifugation, 60.000 g for 30 minutes (~10 kDa)
  - ICP-MS

Result - compost classification

Number of composts in each class

Class	Cd	Cr	Cu	Ni	Pb	Zn	Total
Organic	8	15	6	15	13	9	5
0	2	13	5	13	12	4	1
I	7	2	5	4	4	9	7
II	4	2	7	0	1	4	6
III	3	0	0	0	0	0	3

Result - plant uptake

	Cd		Cr		Cu	
	mg/kg DM	µg/pot	mg/kg	µg/pot	mg/kg	µg/pot
Control	**0.14**	**0.07**	**0.21**	**0.11**	**5.8**	**3.2**
Compost	**0.14**	**0.17**	**0.22**	**0.25**	**6.7**	**7.7**
(min-max)	(0.09-0.25)	(0.10-0.32)	(0.12-0.60)	(0.12-0.77)	(5.1-8.9)	(5.1-12.6)

	Ni		Pb		Zn	
Control	**1.09**	**0.61**	**0.03**	**0.02**	**22.5**	**12.6**
Compost	**1.03**	**1.19**	**0.06**	**0.07**	**25.1**	**29.0**
(min-max)	(0.61-1.41)	(0.56-2.03)	(LOD-0.32)	(LOD-0.38)	(19.6-34.7)	(19.9-43.4)

Results - correlations

Tab. 1. Correlation between different methods used to determine the total and plant available heavy metal concentration in 17 composts

	Zn			Cu			Ni	
	Tot	**XRF**	**CAT**	**Tot**	**XRF**	**CAT**	**Tot**	**XRF**
XRF	0.927*			0.947*			0.675*	
CAT	0.735*	0.831*		0.797*	0.692*		nd	
10kDa	0.073	-0.228	-0.179	0.644*	0.579*	0.946*	-0.089	0.125

	Cr		Cd		Pb	
	Tot	XRF	Tot	XRF	Tot	XRF
XRF	0.747*		nd		0.894*	
10kDa	-0.264	-0.322	0.978*	nd	-0.016	0.023

* significant (P < 0.05); nd: not determined

Results – Regression coefficient between total plant uptake and the 10kDa method (water)

	Zn		Cu		Ni	
	Tot	10kDa	Tot	10kDa	Tot	10kDa
Plant	26.2	71.5	38.1	66.6	36.5	70.7

	Cr		Cd		Pb	
	Tot	10kDa	Tot	10kDa	Tot	10kDa
Plant	22.6	64.0	97.3	90.3	31.2	22.3

Results - summary

- **None of the compost resulted in toxic heavy metal concentration in plants at any of the 5 harvest times**

Correlation between methods

- **Total**

  Wet digestion method and XRF correlated for all heavy metals (Cd was not determined)

- **Total vs Plant extractable (Cu and Zn)**

  Wet digestion and CAT method correlated for Zn and Cu
  XRF method and CAT method correlated for Zn and Cu

  Wet digestion and 10kDa method correlated for Cu and Cd
  XRF and 10kDa method correlated for Cu (Cd not determined)

- **Plant extractable (Cu and Zn)**
  - CAT and 10kDa correlated for Cu

Results - summary

- **Total vs plant uptake**

  The regression coefficient between the compost total heavy metal concentration and plant uptake was only ”good” for Cd

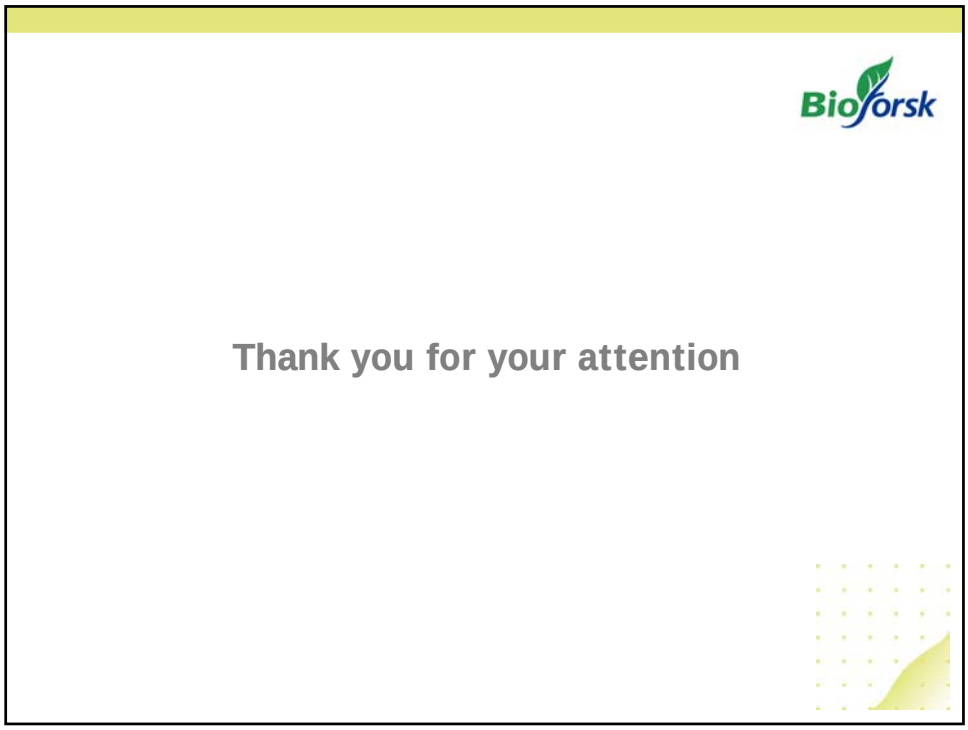
- **10kDa method vs plant uptake**

  Regression coefficients between the 10kDa method and plant uptake were good for Zn, Cu, Ni, Cr and Cd (not for Pb)

Conclusion

- **Determination of the compost heavy metal concentration by XRF can replace the time consuming wet digestion and determination by ICP**
- **Since the wet digestion method and the CAT method correlated, little information on the plant availability is gained by using both methods**
- **The plant availability of heavy metals in compost was best predicted by water extraction (1:10 w/w) for 24 hours and centrifugation at 60.000 g for 30 minutes**
- **Heavy metals attached to small organic molecules are plant available**

Today's method for determination of total heavy metal concentration (wet digestion/ICP) and plant available Cu and Zn (CAT) in composts can easily be changed to XRF analysis (total) and water extracts (plant availability)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