The Effects of Fe-Chelate Type and pH on Substrate Grown Roses

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Abstract

Substrate grown roses appear to be susceptible to chlorosis, which indicates problems with Fe or Mn uptake and hence yield reduction. In common practice this problem is often treated by the addition of extra Fe-chelate, or the use of Fe-EDDHA instead of Fe-DTPA. In previous tests, it was shown that the pH in the root environment is a major factor in the prevention of chlorosis. Moreover, the application of Fe-EDDHA does not always show satisfying improvements in practice. The interaction between Fe-chelate types (EDDHA and DTPA) and pH was studied with roses cv. 'Kiss' and 'Escimo' on glasswool substrate, reusing drainage water. pH levels compared were about 7, 5.8 and 4.5. The treatments resulted in significant chlorosis and consequently yield reduction at high pH with both cultivars and both chelate types. Highest yields were obtained at low pH, especially with 'Escimo'. The Fe uptake was clearly affected by the pH with both chelate types. At high pH the Fe-uptake was significantly higher with Fe-EDDHA; however the Mn contents in the plant were significantly lower with these treatments. The uptake of Zn and Cu was also affected by specific combinations of pH and the type of chelate. It was concluded that an optimal pH control was the best method of preventing chlorosis. The choice of the chelate type was less effective and could enhance Mn deficiency.

INTRODUCTION

Chlorosis is a common phenomenon in substrate grown cut-rose. It is often associated with Fe-deficiency, since there is a strong resemblance with symptoms of Fe deficiency (Winsor and Adams, 1987). This can be caused by high pH in the root environment which is a quite common phenomenon too (Bij de Vaate and Roubos-Hoogstraten, 1997). Actually, pH control in rose appeared to be rather difficult compared to many other substrate grown crops. This is most likely due to the rapid and sequential change in growth stage, following the method of production in flushes, which involves a quite drastic adjustment in the NH_4:NO_3 ratio required in the nutrient supply (Voogt, 1994). In commercial practice these requirements cannot be fulfilled: moreover many growers have a negative attitude towards this necessary high NH_4 supply in the nutrient solution. Fe-DTPA is the standard recommended chelate fertilizer in nutrient solutions for substrate culture (De Kreij et al., 1993). Since chlorosis became an increasing problem, Fe-EDDHA became more and more recommended as it has a much higher stability constant at high pH than Fe-DTPA. Fe-EDDHA is also widely used to cure chlorosis in calcareous soils (Alvarez- Fernadez et al., 2005) and has proven to be effective. However in rose crops, the application of Fe-EDDHA has not always been effective in curing Fe-chlorosis. This study compares two chelate types at a range of pH levels, to investigate the effect on chlorosis, yield, stem quality and (micro-) nutrient uptake.

MATERIALS AND METHODS

Experimental Design

The experiment consisted of three factors: two Fe-chelate types and three pH levels in the nutrient solution, and two rose cultivars. The nutrient solution treatments were laid out in four parallels in two randomized blocks in a greenhouse, and the rose cultivars were laid out as split-plots in each field. Each nutrient treatment consisted of a

closed system, with one supply tank and a central drainage collection for all parallel fields. The cultivars were, 'Escimo' and 'Kiss': in commercial practice known as susceptible and moderately susceptible for chlorosis respectively. The chelate types: Fe-DTPA (AKZO, dissolvine D-FE-6-P®, 6% Fe) and Fe-EDDHA (AKZO, dissolvine Q-Fe-6®, 6% Fe) were used. In all treatments, the target value was 25 µmol L^{-1} Fe in the circulating nutrient solution. The Fe treatments were achieved by operating at high circulation rates as well as weekly analysis of the nutrient solutions and subsequent adjustment of the Fe supply. The pH levels were 7.0, 5.8 and 4.5 in the root environment respectively, basically regulated by the addition of 0.25, 0.75 and 1.25 mmol L^{-1} NH_4 respectively in the standard solution. These concentrations were slightly adjusted depending on the pH developments in time. Fine tuning was done by automatic pH control and subsequent adjustment dosing of either $KHCO_3$ or a mixture of HNO_3 and H_2SO_4.

Growing System

The experiment was conducted in a modern standard greenhouse with growing conditions in accordance with rose growing in commercial practice. Glasswool was chosen as the substrate since it is inert for both pH as well nutrients. Slabs, 20 cm in width and 10 cm in height were wrapped in polythene sheets, provided with drainage slits at 2 cm from the bottom, placed in gutters and covered with black and white polythene sheets. Each individual plant was provided with a trickle nozzle. Drainage water was collected and reused, without sterilization treatment. Irrigation frequency was adjusted to the crop demand, aiming at a drainage rate of 0.8. The base composition of the nutrient solution supplied, for the nutrients not under investigation was: K 5.0, Ca 3.5, Mg 0.75, NO_3 11, SO_4 1.25, P 1.25 all in mmol L^{-1} , Mn 5, Zn 3.5, B, 20 m, Cu 0.75, Mo 0.5 all in µmol L^{-1}. The water source was rainwater, which contained on average 2.5 µmol L^{-1} of Zn, which was taken into account for the Zn supply by the fertilizer recipe. Five-week old plants, grafted on 'Inermis' rootstock, raised in rockwool cubes of 5 * 5 * 7.5 cm were placed on the substrate early March, The experiment was continued for 14 months.

Observations

Crop development was monitored throughout the experiment. Appearance of chlorosis was visually judged six times, using an index range from 0 (none) to 10 (very severe). The yield was observed 6 days a week, with determination of number, weight and length of stems. Flower stem quality, determined by flower bud height and flower colour (visually), was observed at random checks. The vase life was determined twice, using 20 stems per plot from one harvesting date.

Analysis

Samples from the nutrient solution were taken from the collection tank and brought to the laboratory immediately. The samples were filtered using Millipore ceramic filters; the filtrate was acidified with nitric acid to pH 1.0, then analysed by atomic absorption spectroscopy.

Leaf samples were taken three times throughout the trial. "Young laminae" were sampled from shoots due to be harvested within some days, being the first two complete leaves counted from the bottom of the shoot. "Old laminae" were taken from shoots bowed-in, below the cutting zone and were not well defined of age. The leaves were kept cold and sent to the laboratory. Afterwards, the leaves were washed following the procedure of Sonneveld and van Dijk (1982) and then dried at 65-75°C for 24 h. The dry weight was determined, the dried samples were ground with a titanium rotor and after dry digestion in a muffle furnace (480°C) the ashes were dissolved using HCl. The elements Fe, Mn, Cu and Zn were determined using an atomic absorption Perkin-Elmer 4000 spectrophotometer.

Chelate Stability

Calculations of the stability of the association of the metal ions with the chelate complex were carried by AKZO-NOBEL with the model MINEQL (Reichwein, 2007).

RESULTS

Chlorosis

Ten weeks after planting the first chlorosis symptoms appeared, mainly at the highest pH treatments and the symptoms were more severe with 'Escimo' than with 'Kiss' (Table 1). During summer and autumn, the chlorosis increased at the high pH level and disappeared more or less at the mid-range and the low pH. At high pH in the first three months the chlorosis was more severe with DTPA than with EDDHA; however, later on no differences were visible between the two chelate types. The differences between the two cultivars were negligible eventually. Severe leaf senescence and leaf dropping appeared 5 months after planting in the treatment with EDDHA - high pH. This began with complete yellowing and necrotic spots on the leaves, with complete necrotic leaves eventually. Tissue analyses confirmed the supposition that this was caused by Mn deficiency.

Yield and Quality

The yield of the first flush of flowers, up to June 15th was not significantly affected by the treatments (Table 2). However by October 1st, the yield was 20-25% lower at the high pH levels, which increased to 30-40% lower yields at the end of the trial. The yield at the lowest pH levels was significantly higher than with both the mid-range and high pH levels, except for the treatment with EDDHA and cv. Kiss. The pH effect on yield was greater for EDDHA than for DTPA, with both cultivars. Apart from the interaction with pH, there were no significant yield effects between the two chelate types. In addition to the effect on production, the stem quality, expressed as stem weight and length was negatively affected also by the high pH (Table 2), with both cultivars. There was no difference in stem quality between mid-range and low pH. No significant differences were found between the treatments in flower bud size, bud colour and vase life of stems.

Nutrient Solution

The intended pH levels in the running nutrient solution were more or less achieved (Table 3). However, sometimes there were strong fluctuations and on average the pH at both the two high levels was somewhat above 7. The estimated NH_4 concentrations in the supply have been broadly achieved. However, with the low pH a substantial quantity of HCO_3^- was necessary to achieve the desired value. During the experiment maintaining the Fe concentration at the target value proved difficult. On average the Fe concentrations were 20-30% higher than intended (Table 4). The actual Fe concentration supplied, expressed in μmol Fe L^{-1} of the net water consumption of the crop is much lower than the average concentration in the root environment and differs strongly among the treatments. With increasing pH, more chelate was required: this need was greater at the lowest pH values. A remarkable interaction between the treatments and the Mn and Zn concentrations in the root environment was evident. With increasing pH, substantially more Mn has been supplied, but despite this, the Mn concentration in the root environment was reduced, except for DTPA at the highest pH. Zn accumulated strongly with DTPA and increasing pH, whilst with EDDHA this was opposite, even with an increased supply of this element (Table 4).

Tissue Analysis

The Fe contents increased with decreasing pH in both young and old laminae and with both cultivars (Table 5). At the highest pH, the Fe contents were 20-30% higher with EDDHA compared to DTPA. Mn and Zn contents increased also with decreasing pH.

However, with Mn, the content was extremely low with EDDHA at the highest pH level. With EDDHA the Zn contents were overall slightly higher than with DTPA. There were no significant differences in Fe, Mn and Zn contents between the two cultivars. For Cu, there was a tendency towards increasing contents with decreasing pH., for DTPA and 'Kiss' in particular, whilst with EDDHA and 'Escimo' the differences were negligible. For Mo, the effect is opposite to the other micro elements ie low pH causes reduced uptake, which is a rather well-known phenomenon (Marschner, 1995).

DISCUSSION

Not surprisingly, the pH was the main factor inducing chlorosis symptoms. The differences between DTPA and EDDHA were very small: chlorosis with EDDHA was less than with DTPA only in the first weeks after planting. The cultivars 'Kiss' and 'Escimo' were equally susceptible. The severe chlorosis coincides with the yield reduction at high pH, and is connected with the reduced Fe and Mn uptake. The differences in the incidence of chlorosis are much more apparent than those in Fe content and show even a better relationship with the Mn content. However it is hard to say whether the observed chlorosis is caused by either Fe or Mn deficiency, or by both, since the symptoms are quite similar (White, 1972).

At the lowest pH the yield was still significantly higher compared to the mid-range pH and cannot be explained by differences in chlorosis incidence only. The differences in Fe content between pH 5.8 and 4.5 are negligible, so the positive yield response with decreasing pH can probably not be explained by improved Fe uptake only. The positive effect of decreased pH on rose and other chlorosis-susceptible crops was found in previous experiments (Voogt, 1994, 1995; Sonneveld and Voogt, 1997) and often associated with increased micro element uptake. Nevertheless it is still not quite clear if this is the only explanation.

The effects of the treatments on micro nutrient uptake and hence on yield and chlorosis can be explained by the thermodynamic stability of the ion-chelate complex. The so- called stability constants, which express the degree of affinity of a metal ion to a complex are derived from the equilibrium of a metal ion and the association of this ion to its chelating compound. The stability constant for a chelating agent is specific for each individual metal ion and depends also largely on the pH (Bugter and Reichwein, 2007), so in mixed nutrient solutions substitution of ions will occur with changing pH. From laboratory tests, the relative stability of a specific metal-chelate complex, related to the total quantity chelated can be computed for mixed solution under different pH values, taking into account also other chemical processes as oxidation and precipitation. The results of such model calculations are performed by MINEQL and presented in Figure 1 (Reichwein, 2007).

The better performance of EDDHA in relation to the Fe uptake at high pH is obviously related to the high affinity for Fe over a broad pH range. For DTPA the affinity for Fe becomes lower than that for Cu at pH 5.0 and for Zn at pH>6.5. The complexation of ZnDTPA might explain the Zn accumulation in the root environment with high pH (Table 4). Nevertheless, the Zn uptake is not increased with increasing pH but is even lower compared to the lower pH treatments. It is suggested that chelated Zn is less or even not unavailable for plants. This phenomenon has been found in previous experiments (Voogt, unpublished data).

The differences in Cu uptake, cannot be explained easily. The affinity for Cu is higher than for Fe, both for EDDHA over a broad pH range and for DTPA at pH>5, so it is plausible that all Cu^{2+} ions are chelated in the root environment. Nevertheless there is a definite decrease in Cu uptake with decreasing pH, especially with DTPA. For the specific DTPA – pH 4.5 treatment, it may be concluded that the availability of free Cu^{2+} ions, is less effective than chelated Cu. However this assumption has no effect for the higher pH values.

The differences in Mn uptake between pH and chelate treatments can be explained by both the stability constants and oxidization processes involved. The Mn^{2+} supplied will

be oxidized to Mn^{3+} or Mn^{4+}, by bacterial activity, followed by rapid precipitation as MnO_2 (Bromfield, 1978), making Mn unavailable to the plant. This process will start after some time, when bacteria will colonize the growing medium, especially with pH values above 6.5. The supply from the stock solution will provide some Mn^{2+} in the solution throughout the experiment. At pH>7.5 with DTPA this ion will be partly chelated due to its higher stability compared to Fe^{3+} (Fig. 1) and thus protected from further oxidation. However, with EDDHA the affinity with Mn^{2+} is almost zero at all pH levels. Therefore Mn^{2+} is liable to be completely oxidized, making it unavailable to the plant. The assumption that the severe chlorosis and the severe leaf drop at treatment 4 (EDDHA and high pH) was caused by Mn deficiency was confirmed by additional specific leaf samples taken for this purpose. Mn contents in laminae with severe symptoms were 0.04 mmol kg^{-1} dry matter.

So, to prevent and cure chlorosis, changing pH proved to be much more effective than supplementation with chelate. However manipulation of the pH is not easy as is shown in earlier studies and also this experiment: major fluctuations in pH can occur and relatively large quantities of acid and base are required for correction of pH (Table 3). So for commercial practice the recommendation is to supply Fe-EDDHA if pH is high. However given the fact that with increasing pH, the Mn availability is reduced drastically, pH>6.5 should be avoided.

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Tables

Table 1. Chlorosis index (visually judged 1=no chlorosis, 10=severe), average of 7 observations during the experiment.

No.	Treatment		Chlorosis index	
	Fe-chelate	pH	'Kiss'	'Escimo'
1	DTPA	7.0	6.6	6.9
2	DTPA	5.8	3.8	4.1
3	DTPA	4.5	4.3	4.4
4	EDDHA	7.0	6.4	7.0
5	EDDHA	5.8	4.2	4.6
6	EDDHA	4.5	4.0	4.5

Table 2. Stems m^{-2} of the first flush of flowers (15 June) and the total number of stems, the average stem weight and the stem length (April 30th).

Treatment		Stems m^{-2} 15 June		Stems m^{-2} 30- Apr		Stem weight (g)		Stem length (cm)	
Fe-chelate	pH	'Kiss'	'Escimo'	'Kiss'	'Escimo'	'Kiss'	'Escimo'	'Kiss'	'Escimo'
DTPA	7.0	38^{a}	28^{a}	181^{b}	182^{b}	16.7^{a}	15.2^{a}	62.0^{a}	61.3^{a}
DTPA	5.8	37^{a}	25^{a}	255^{c}	271^{c}	18.6^{b}	17.2^{b}	66.4^{b}	65.9^{b}
DTPA	4.5	41^{a}	32^{a}	266^{d}	293^{d}	18.3^{b}	15.9^{c}	66.4^{b}	64.2^{c}
EDDHA	7.0	32^{a}	29^{a}	174^{a}	164^{a}	17.4^{a}	16.1^{c}	66.3^{b}	65.1^{bc}
EDDHA	5.8	31^{a}	31^{a}	260^{cd}	281^{c}	17.4^{a}	16.5^{bc}	65.6^{b}	65.5^{b}
EDDHA	4.5	33^{a}	32^{a}	258^{cd}	298^{d}	19.5^{c}	16.4^{bc}	66.9^{b}	65.1^{bc}

Results with equal letters within a column do not differ significantly from each other ($P<0.05$).

Table 3. Average pH in the root environment and the supply of additional NH_4^+ in the nutrient solution and acid and base by the automatic pH control.

No.	Treatment			Supply (mmol L^{-1})		
	Fe-chelate	pH target	pH measured	NH_4^+	H^+	HCO_3^-
1	DTPA	7.0	7.3	0.1	0.2	0
2	DTPA	5.8	5.7	0.8	0.9	1
3	DTPA	4.5	4.4	1.2	0.3	2.2
4	EDDHA	7.0	7.2	0.1	0.1	0
5	EDDHA	5.8	5.6	0.8	0.9	1.4
6	EDDHA	4.5	4.4	1.2	0.6	2.2

Table 4. Total supply of Fe, Mn and Zn, expressed per L water absorbed by the crop, and the average concentrations in the root environment.

Treatment		Supply (µmol L^{-1})				Root environment (µmol L^{-1})			
Chelate	pH	Fe	Mn	Zn	Cu	Fe	Mn	Zn	Cu
DTPA	7.0	21	15	2.5	0.8	26	3.5	21	2.5
DTPA	5.8	14	8	2.5	0.8	31	1.9	14	1.8
DTPA	4.5	9	5	2.5	0.8	39	3.6	9	1.3
EDDHA	7.0	16	18	5	0.8	30	0.4	2	1.8
EDDHA	5.8	12	10	4	0.8	32	2.4	5	1.5
EDDHA	4.5	11	5	3.5	0.8	33	3.3	6	1.3

Table 5. Micronutrient contents in young fully grown laminae, (average of three samples).

No.	Treatment			Fe	Mn	Zn	B	Cu	Mo
	Fe-chelate	pH level		(mmol kg^{-1} dry matter)				(µmol kg^{-1} d.m.)	
1	DTPA	7.0	'Kiss'	0.92	0.42	0.23	4.28	28	21
2	DTPA	5.8	'Kiss'	1.25	1.31	0.41	4.53	74	17
3	DTPA	4.5	'Kiss'	1.39	1.49	0.52	4.05	77	13
4	EDDHA	7.0	'Kiss'	1.16	0.13	0.29	5.50	61	26
5	EDDHA	5.8	'Kiss'	1.22	1.40	0.53	5.02	76	19
6	EDDHA	4.5	'Kiss'	1.30	1.56	0.64	4.49	93	17
1	DTPA	7.0	'Escimo'	0.76	0.40	0.25	4.48	55	19
2	DTPA	5.8	'Escimo'	1.10	1.30	0.42	4.37	83	15
3	DTPA	4.5	'Escimo'	1.33	1.36	0.46	3.66	95	18
4	EDDHA	7.0	'Escimo'	1.10	0.14	0.28	5.31	75	24
5	EDDHA	5.8	'Escimo'	1.20	1.37	0.49	4.87	72	18
6	EDDHA	4.5	'Escimo'	1.34	1.33	0.53	4.12	79	13

Figures

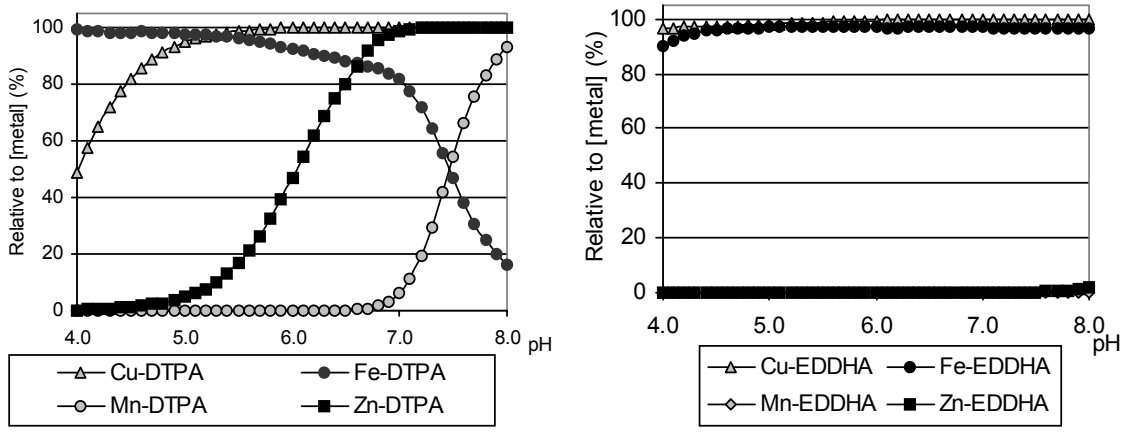

Fig. 1. Relative complexation of metal ions by DTPA and EDDHA in relation with the pH in mixed nutrient solutions, calculated by MINEQL.