Microfibril Angle and Density Patterns of Fertilized and Irrigated Norway Spruce

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Two Norway spruce nutrient optimisation trials, one in the north of Sweden and one in the south, were used to study the effects of intensive growth and fertilization on wood density and microfibril angle. Three different treatments and a control were available; daily irrigation, daily liquid fertilization and solid fertilization. The nutrient optimisation was based on foliage analysis and the solid fertilization essentially comprised the same amount of nutrients but was applied annually in solid form. Measurements of density and microfibril angle (MFA) were performed using X-ray diffraction. Growth rate, expressed as a transformation of annual ring width, was very important at the southern site when the effect of cambial maturation had been taken into account. Effects of both fertilization and irrigation remained strong and significant for density, and irrigation was a significant factor explaining MFA. At the northern site distance from pith was the dominant factor but the effect of growth rate was also strong and the treatment effect was significant for both density and MFA. The combination of higher MFA and decrease in density for fertilized trees resulted in a lower calculated strength of the wood. An over 100% increase in ring width only corresponded to approximately a 20% decrease in wood density and the production of wood dry matter was hence increased by treatments.

Keywords Ring width, growth rate, MoE, X-ray diffraction, juvenile wood
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1 Introduction

In Sweden, the application of a more intensive silvicultural management on selected areas, is being investigated. Such a regime would include planting of selected material, repeated fertilizations and shorter rotations. However, the effect of the accompanying increase in volume production on wood properties, and the subsequent utilisation of the resource, is a cause of uncertainty. Wood properties that can be used as predictors of product properties include wood density and microfibril angle. These properties, on their own or together, are common predictors of wood and fibre shrinkage, stiffness and strength and are therefore important for pulp and paper production as well as for solid wood utilisation.

Density, a compound property, is a measure of the amount of cell wall material in the wood and depends on the thickness of the cell wall and cell width, and on a broader scale, the relative proportions of latewood and earlywood; the density of spruce decreasing with an increase in growth rate (Olesen 1976, Zobel and Van Buijtenen 1989, Simpson and Denne 1997). The density of the wood impacts the pulping process, energy consumption and is important for pulp yield (Varhimo and Tuovinen 1999). High density had a negative correlation with optical properties and a positive correlation with tear strength but the effects diminished when position in the tree was taken into account indicating that it is not the density in itself that is important but rather the properties of the fibres which are mirrored by the density (Braaten 1996). The stiffness and strength of solid wood is positively correlated with density (Cown et al. 1999). Wood density has been reported to account for 70% of the variation in modulus of elasticity (MoE) and microfibril angle for 86%: and these two properties have been successfully combined to predict the MoE of small specimens (Evans and Ilic 2001).

The microfibril angle is the angle between the cellulose fibrils and the fibre axis. As microfibrils can only shrink transversely, a large angle implies increased longitudinal shrinkage of the tracheid and consequently increased longitudinal shrinkage of the wood. Wood stiffness and bending strength are negatively affected by large microfibril angles (Cave and Walker 1994); the tensile strength of the tracheid decreasing with increasing angle (Kellog and Thykeson 1975, Armstrong et al. 1977, Page et al. 1977). High microfibril angle has been shown to cause low tear strength of paper (Uprichard et al. 1994). Juvenile- and compression wood are known to have higher microfibril angles than other woods (Donaldson 1992, 1996, Gorisek et al 1999).

Wood properties change from pith and out and the largest changes occur within the juvenile wood which is the inner part of the stem. A general pattern of density decrease immidiately adjacent to the pith followed by an increase towards bark which later diminishes is well established for Norway spruce (Olesen 1976, Johansson 1993, Danborg 1994, Pape 1999). The radial pattern of high microfibril angle near the pith, and the decrease and stabilisation towards a lower value for more mature wood has also been well described (McMillin 1973, Donaldson 1992, Shupe 1996, Lindström et al. 1998, Saranpää et al. 2000).

The application of fertilizers, especially nitrogen, can cause formation of wood with lower density (Shepard 1985, Mäkinen et al. 2002). An increase in growth rate due to fertilization is reported to change wood properties more than a corresponding increase due to for example thinning (Zobel and van Buijtenen 1989); however, the decrease in density is generally outweighed by the relative increase in volume production, which renders a larger dry matter production. The positive effects of growth rate on microfibril angle, both for overall development and development within rings, has been ascertained for Norway spruce (Lindström et al. 1998, Herman et al. 1999, Saranpää et al. 2000). The nominal effects of increased growth rates are usually small compared with the decrease that occurs within the juvenile core. In a study on fertilized Douglas-fir, 95% of the variation in microfibril angle was accounted for by age, whereas an increase in growth rate accounted for only a few percent (Erickson and Arima 1974).

The aim of this study was to study the effect of fertilization and irrigation on density and microfibril angle on Norway spruce (*Picea abies* (L.) Karst.). Nutrient optimisation trials with very high growth and volume responses were used to establish an upper limit to wood property responses. The development from pith and out was analysed in relation to effects due to growth and treatment.

2 Methods

2.1 Data

The data; site, sampling and sample preparation are described in Lundgren (2004). The wood samples were conditioned to 20°C and 40% relative humidity. All variables were measured under these conditions which correspond to a moisture content of approximately 8% (Keylwerth 1969). The measured density will be denoted as density* in this paper to indicate that it is not the basic density.

The samples were run in the SilviScan machine to obtain microfibril angle and density. X-ray diffraction was used for density measurements and the resulting diffraction pattern was used to measure the microfibril angle (MFA). Measurement points for density were obtained every 0.05 mm, and for MFA the means over distances of 5 mm were obtained. Averages per annual ring were calculated for all variables.

2.2 Analyses

In order to facilitate comparisons between the density at 8% and the more commonly used basic density, a transformation, based on average sizes of wood samples for the different sites and treatments, was performed. The calculation was based on average size of wood samples for the different sites and treatments. Radial shrinkage was assumed to be 3% and tangential shrinkage 6% from a 30% moisture content. Dry weight was divided by raw volume to obtain an estimate of basic density. Modulus of elasticity (MoE) was estimated using measured density, MFA and the empirical formulas given by Evans and Ilic (2001).

Initial analyses also confirmed that neither plot nor block did have an effect; thus, those effects were not included in the model. Density, MFA and MoE were plotted by calendar year in order to visually examine the effect of treatments. In order to statistically analyse these effects the development from pith and out due to cambial maturation will have to be considered. Density followed a linear shape by distance from pith for both Asa and Flakaliden (Eq. 1). The declining shape of MFA with distance from pith was taken into account using the inverse of distance from pith (Eq. 2) and MoE was analysed with a quadratic regression (Eq. 3).

$$Y_{ijkl} = \mu + \beta_0 d_k + \beta_2 I_i + \beta_3 F_j + r_k + \varepsilon_{ijkl} \quad (1)$$

$$Y_{ijkl} = \mu + \beta_0 id_k + \beta_1 I_i + \beta_2 F_j + \beta_3 IFid_{ijk} + r_k + n_k + \varepsilon_{ijkl} \quad (2)$$

$$Y_{ijkl} = \mu + \beta_0 d_k + \beta_1 d^2{}_k + \beta_2 I_i + \beta_3 F_j + \beta_4 IFd_{ij} + r_k + n_k + \varepsilon_{ijkl} \quad (3)$$

where Y_{ijkl} represents the dependent variable, μ is the overall mean, $\beta_0 - \beta_4$ are fixed effects for: d – distance from pith, id – inverse of distance from pith, I – irrigation, F – fertilization, r – ring width, n – cambial age (ring number counted from pith) and ε_{ijkl} is random error. The mixed procedure in SAS statistical program package, was used for the analysis (SAS OnlineDoc 2000). A repeated measures analysis of variance was used (k repeated measures per sample) and the growth curve part of the models (d, id and d^2) were included as random components. The covariance matrix was assumed to be unstructured. I and F were treated as dummies representing the presence of irrigation and fertilization and assuming the value 1 or 0 depending on whether the sample came from a treated plot or not and whether the ring had formed prior to or after 1987, the first year of treatment, or not. At Flakaliden where the I treatment was not sampled, the I and F dummies were not used. Instead three levels of T (C, F and IL) were included in the model. In order to avoid discrepancies in the data which may occur close to the pith, rings closer to the pith than 2 rings were excluded from the statistical analysis. Effects that were far from being significant ($p > 0.10$) were not included in the final models except for the effects of treatment which were always included.

In order to assess the combined effect of density and growth, a volume and dry matter calculation was performed. Volume per tree was calculated using breast height diameter and tree height (Brandel 1990). Density at breast height was calculated by weighing annual ring density by the area of the growth ring. Whole-tree densities were estimated with the functions presented by Hakkila (1966). An analysis of variance was performed using Eq. 4.

$$Y_{ij} = \mu + \tau_i + \varepsilon_{ij} \tag{4}$$

Where Y_{ij} , is either volume or dry matter, τ_i, is treatment and ε_{ij} is random error. Due to the smaller sample size the sampled F-trees at Asa were, at the onset of fertilization, on average one year younger than the trees sampled from the other treatments. The model (4) was therefore adjusted by including initial tree size when analysing the Asa data. The SAS routine Proc GLM was used. Least square means were calculated and compared between treatments and significance levels were adjusted for multiple comparisons using the Bonferroni option.

3 Results

The age of the cambium at the start of fertilization ranged from 2–6 years at Asa and from 8–14 years at Flakaliden. The basic density was approximately 15% lower than mean sample density at 8.3% moisture content (Table 1).

Growth rate, expressed as ring width, exceeded the controls for all treatments for both Asa and Flakaliden (Fig. 1). In the last few years of the treatments, the difference diminished and even reversed at Asa in 1998, so that the control had the widest rings. This was due to increased competition in the fertilized plots that also affected the wood properties, which became more similar in the last few rings (Fig. 1–3).

Table 1. Mean sample length and density at 20°C and 40% RH and corresponding calculated basic density.

Site	Treatment	Mean sample length, mm	Mean sample density, kgm^{-3}	Calculated basic density, kgm^{-3}
Asa	C	53.3	415.5	350.9
	I	54.0	439.0	370.7
	F	54.1	397.6	335.8
	IL	66.9	382.5	323.0
Flakaliden	C	38.2	474.5	400.7
	F	61.0	403.0	340.3.
	IL	69.9	397.8	336.0

Table 2. Mixed model analysis of variance, type III tests of fixed effects at Asa.

Effect	Density F	Density p	MFA F	MFA p	MoE F	MoE p
I	15.70	< 0.0001	7.81	0.0053	12.55	0.0004
F	29.41	< 0.0001	2.03	0.1544	5.11	0.0241
d	32.70	< 0.0001	–	–	14.72	0.0003
d^2	–	–	–	–	33.17	< 0.0001
id	–	–	65.05	< 0.0001	–	–
IFd	–	–	–	–	11.03	< 0.0001
IFid	–	–	3.44	0.0165	–	–
r	384.79	< 0.0001	137.63	< 0.0001	210.83	< 0.0001
n	–	–	5.48	0.0195	53.42	< 0.0001

Table 3. Mixed model analysis of variance, type III tests of fixed effects at Flakaliden.

Effect	Density F	Density p	MFA F	MFA p	MoE F	MoE p
T	7.44	0.0006	22.41	< 0.0001	17.40	< 0.0001
d	221.35	< 0.0001	–	–	165.31	< 0.0001
d^2	–	–	–	–	99.84	< 0.0001
id	–	–	193.63	< 0.0001	–	–
Td	–	–	–	–	15.81	< 0.0001
Td^2	–	–	–	–	10.66	< 0.0001
r	186.62	< 0.0001	5.56	< 0.0001	191.01	< 0.0001

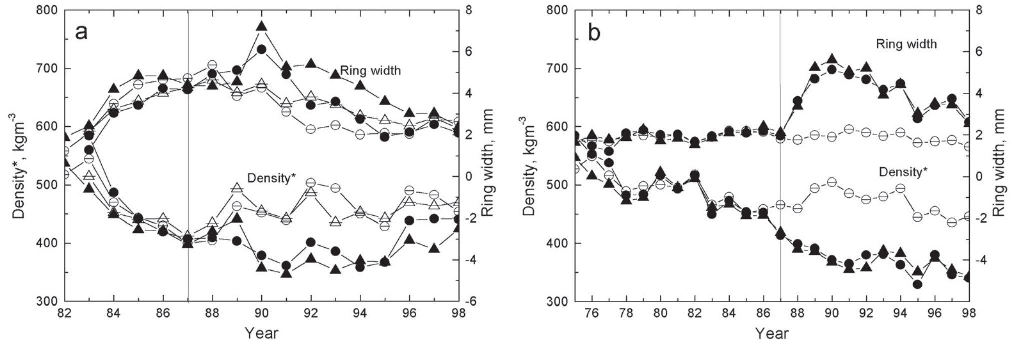

Fig. 1. Annual ring width and density*, mean values per year for a) Asa and b) Flakaliden. Vertical dotted line indicates commencement of treatments ○ = C, control, △ = I, irrigation, ● = F, solid fertilization and ▲ = IL, liquid fertilization. Density* denotes density at 8% moisture content.

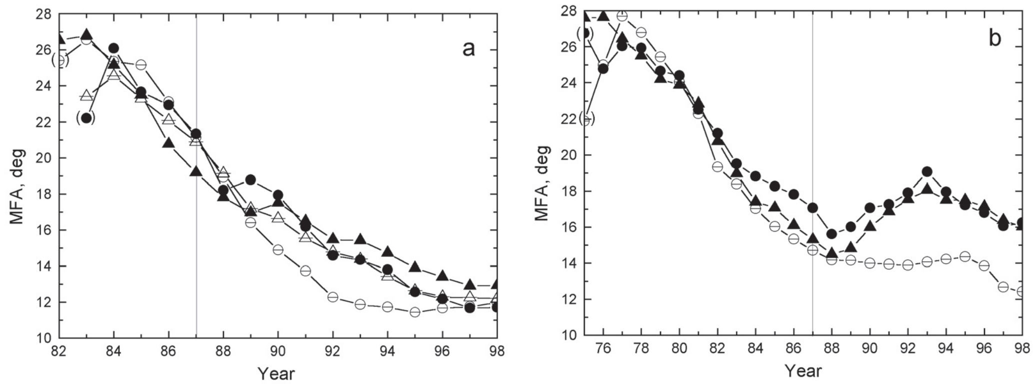

Fig. 2. MFA, mean values per year for a) Asa and b) Flakaliden. Vertical dotted line indicates commencement of treatments ○ = C, control, △ = I, irrigation, ● = F, solid fertilization and ▲ = IL, liquid fertilization.

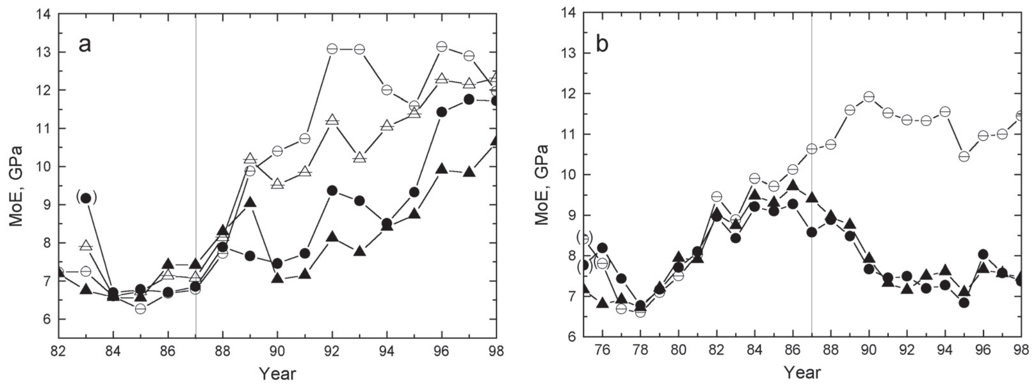

Fig. 3. Calculated MOE, mean values per year for a) Asa and b) Flakaliden. Vertical dotted line indicates commencement of treatments ○ = C, control, △ = I, irrigation, ● = F, solid fertilization and ▲ = IL, liquid fertilization.

The mixed model analysis of variance revealed significant effects of fertilization and irrigation on density at Asa (Table 2). At Flakaliden treatment had a significant effect on density but the most important factor was distance from pith (Table 3). Density decreased with distance from pith (Tables 4 and 5, Fig. 4) indicating that the bulk of the samples are still within the juvenile

Table 4. Models for fixed effects at Asa.

Effect	I	F	Density Estimate	Density s.e.	MFA Estimate	MFA s.e.	MoE Estimate	MoE s.e.
Int.			526	8.70	10.5	1.03	9.01	0.469
I	0		–19.1	4.83	–1.10	0.394	–1.24	0.349
I	1		0	.	0	.	0	.
F		0	29.1	5.37	–0.575	0.403	–0.912	0.403
F		1	0	.	0	.	0	.
d			–0.818	0.143	–	–	0.0472	0.0251
d^2			–	–	–	–	–1.96E-3	3.39E-4
id			–	–	59.5	16.6	–	–
IFd	0	0	–	–	–	–	0.0912	0.0160
IFd	0	1	–	–	–	–	0.0306	0.0148
IFd	1	0	–	–	–	–	0.0420	0.0142
IFd	1	1	–	–	–	–	0	.
IFid	0	0	–	–	45.9	14.7	–	–
IFid	0	1	–	–	36.0	18.3	–	–
IFid	1	0	–	–	42.3	16.1	–	–
IFid	1	1	–	–	0	.	–	–
r			–21.1	1.08	0.923	0.0786	–0.634	0.0438
n			–	–	–0.119	0.0508	0.420	0.057

Table 5. Models for fixed effects at Flakaliden.

Effect	T I	Density Estimate	Density s.e.	MFA Estimate	MFA s.e.	MoE Estimate	MoE s.e.
Int.		521	4.48	12.1	0.542	7.51	0.204
T	C	19.1	4.96	–2.03	0.496	–2.01	0.358
T	F	7.24	4.89	1.27	0.401	–0.204	0.301
T	IL	0	.	0	.	0	.
d		–1.87	0.126	–	–	0.209	0.0323
d^2		–	–	–	–	–0.0290	5.51E-4
id		–	–	90.0	6.47	–	–
Td	C	–	–	–	–	0.219	0.0497
Td	F	–	–	–	–	–0.054	0.0435
Td	IL	–	–	–	–	0	.
Td^2	C	–	–	–	–	–3.52E-3	9.49E-4
Td^2	F	–	–	–	–	8.54E-4	7.77E-4
Td^2	IL	–	–	–	–	0	.
r		–16.0	1.17	0.605	0.111	–0.650	0.0470

phase of wood formation. At Flakaliden the density response appeared already in the first year of treatment even though the response in ring width not occured until the year after (Fig. 1b). The density profile for an IL sample from Flakaliden (Fig. 6.) shows that the density peak of the latewood in 1987 was smaller than the preceding years.

At Asa MFA was most strongly influenced by ring width (Table 2). Irrigation but not fertilization had a significant effect. At Flakaliden the distance factor was more important (Table 3). The development by year for ring width and MFA at Flakaliden (Fig. 1b and 2b) showed a peak in ring width in 1990 and a peak in MFA in 1993 which may indicate a delay in the effect of ring width.

For MoE, which is a combination of density and MFA, the pattern that growth rate was comparably more important at Asa and that the development from pith and out had a stronger influence at Flakaliden persisted. At Asa the irrigation factor was significant which is supported by the differences between the C and I curves in Figs. 2a and 3a.

The density of control trees was at a similar level

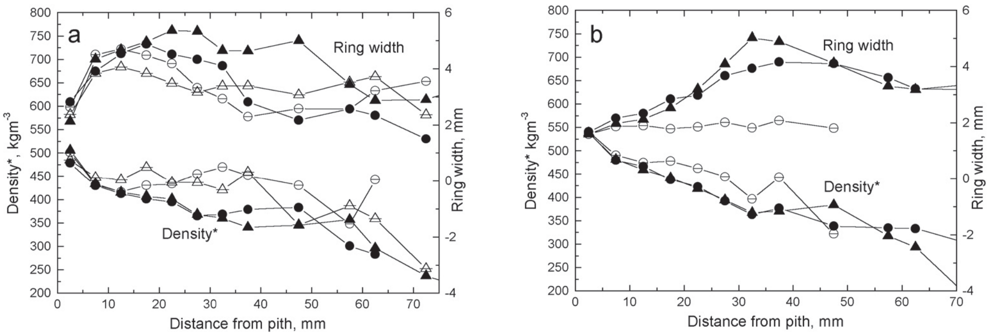

Fig. 4. Mean annual ring width and density* by distance from pith a) Asa and b) Flakaliden. ○ = C, control, △ = I, irrigation, ● = F, solid fertilization and ▲ = IL, liquid fertilization. Density* denotes density at 8% moisture content.

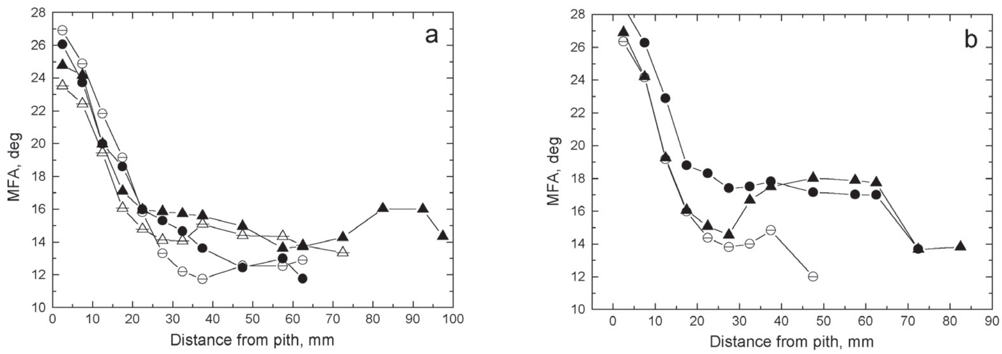

Fig. 5. Mean MFA by distance from pith a) Asa and b) Flakaliden. ○ = C, control, △ = I, irrigation, ● = F, solid fertilization and ▲ = IL, liquid fertilization.

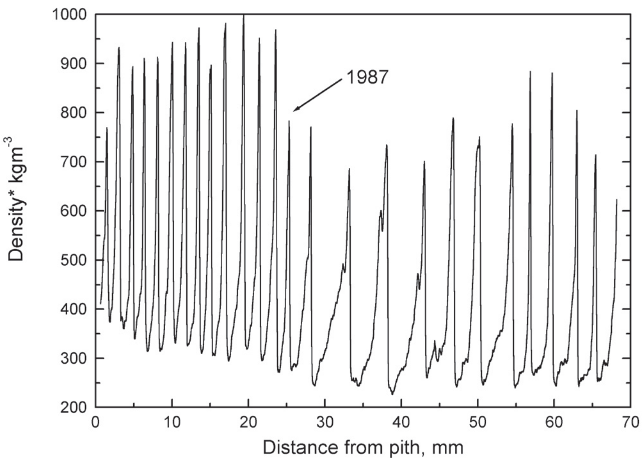

Fig. 6. Density profile for an IL sample from Flakaliden, year of commencement of treatments is indicated.

at Asa and Flakaliden (varied around 550 kgm^{-3}) in the last 10 years (Fig. 1). The level of MFA, which in Fig. 2 appears to have reached a stable minimum at least at Asa, is higher at Flakaliden which is why the calculated MoE is lower.

Single tree volume was significantly affected by treatment at both Asa ($F = 8.98$, $p = 0.0001$) and Flakaliden ($F = 25.78$, $p < 0.0001$). At Asa the least square means volume (Table 7) of IL was significantly different from C ($p < 0.0001$) and I ($p = 0.0364$). At Flakaliden C differed significantly from F and IL (both $p < 0.0001$). Dry matter was also significantly affected by treatment at both Asa ($F = 6.92$, $p = 0.0007$) and Flakaliden ($F = 21.31$, $p < 0.0001$). At Asa the least square means of dry matter differed between C and IL ($p= 0.0003$). At Flakaliden C differed from F ($p=0.0002$) and IL ($p<0.0001$). The differences in volume produced are hence slightly offset by the change in density which is why the effect on dry matter production is somewhat weaker but still substantial (Table 7).

Table 6. Covariance parameter estimates.

	Density *d*	MFA *id*	MoE *d*	MoE *d*^{2}
Asa	0.4566	2844.78	0.0108	3.77E-6
Flakaliden	0.4947	1883.68	0.0141	2.40E-4

Table 7. Least square means of tree volume and tree dry matter.

τ_i,	Asa* Volume, dm^3	Asa* Dry matter, kg	Flakaliden Volume, dm^3	Flakaliden Dry matter kg
C	66.0	23.2	21.3	7.8
F	99.8	31.9	61.2	18.8
I	90.8	31.2	–	–
IL	123.7	37.9	77.7	23.9

* Least square means adjusted for initial tree size

4 Discussion

The absolute treatment effect on density and MFA was greater at Flakaliden in the north, which was a poorer site with a larger response in growth (Fig. 1 and Fig. 2). The untreated controls displayed lower densities and higher MFA's at Flakaliden than at Asa. The effect of fertilization on ring width and density was quite distinct in the material studied. Fertilization caused formation of wood with lower density, which is in line with previous findings (Klem 1972, Mäkinen 2002). Mäkinen, studying partly the same trials (C and IL at Flakaliden) also noted that the density was steadily decreasing and hence still in its juvenile phase. The findings in the present paper agrees with that; parameter estimates of distance from pith are negative. Fertilization and irrigation are known to adversely affect latewood ratio due to a prolonged formation of earlywood (Brix 1972, Kao Hsu and Walters 1975). In the present study, the response in density was immediate the first year of treatment whereas the response in ring width did not start until the second year at Flakaliden (Fig. 1b), which indeed appears to have been an effect of very little latewood in the first year of fertilization (Fig. 6).

The general pattern of MFA and the response of MFA to growth rate agrees with the findings of Lindström et al. (1998) and Saranpää et al. (2000). The findings presented here indicated that treatments which produces noticeable effects on growth may have an undesired impact on microfibril angle development. The MFA's for the irrigated trees at Asa were almost as large as the trees subjected to nutrient optimisation (Fig. 2a and Fig. 4a). This pattern cannot fully be explained by ring width (Fig. 1a) but previous studies on the material have shown that irrigation does have an effect on the volume production at this site (Bergh et al 1999). As a result the wood formed can be expected to be weaker than normal control wood.

The microfibril angle of earlywood tracheids is higher than that of latewood tracheids (McMillin 1973) and a development of steadily decreasing microfibril angle across the annual ring has been shown for Norway spruce both within the earlywood (Bergander et al. 2002) as well as across the whole ring from early- to latewood (Herman et al. 1999). The latter study also shows that rings from fast growing trees have consistently larger microfibril angles throughout the rings. The effect of growth rate on MFA shown in the present paper were thus likely to have been an affect of both varying relationship between high-MFA earlywood and low MFA-latewood as well as of a higher level of MFA across the whole ring. The apparent lagged effect of ring width on MFA which was observed at Flakaliden (Figs. 1b and 2b) can explain that effects of growth rates at this site (Table 3) were comparably weak.

Even though the analyses from the Flakaliden site contained no irrigation treatment, the differences in effects between the two sites can be compared as all analyses for Asa were run both with and without the irrigation with similar results. However, other differences between the sites need to be considered. Firstly, the treatments at Flakaliden coincided with the cessation of juvenile wood formation as far as MFA is concerned, but the treatments at Asa started a few years before the MFA curve flattened out. Secondly, trees in the treated plots at Asa had reached a more severe competitive state in the later period of the study, whereas the IL and F plots at Flakaliden had just started to be affected by competition. The trees at Asa reached a lower stable level of MFA faster than those at Flakaliden. The distance from pith-plots (Figs. 4 and 5) indicates that this material was still within its juvenile wood development phase in 1999 when they were sampled. The results, and in particular the models presented, (Tables 4, 5 and 6) should therefore be interpreted and used with that in mind. In a traditional silvicultural regime the mature wood would be the wood formed after fertilization at least at breast height, and this outer wood would also be the wood which would end up at the pulpmill. The possible future analysis of the effect of nutrient optimisation on mature wood will in this material be obscured by the increase in competition which at Asa was already quite apparent in the last few years (Fig. 1a).

The MoE calculations should be regarded as a theoretical illustration of the combined effects of an increase in MFA and decrease in density of wood stiffness of fertilized stands. The calculated levels appeared reasonable compared with existing findings from Sweden were MoE of spruce is said to range between 8.3 and 13 GPa (Boutelje and Rydell 1986) and Norway, where MoE of 9.7 GPa has been measured for clear test specimens (Okstad and Karstad 1985). The stiffness of the wood was adversely affected by the fertilization and the wood produced might not qualify as high-grade structural timber. The stiffness values presented in this work are predictions for clear wood and other factors such as the effect of knots need to be considered to determine how boards sawn from these trees will perform. Mäkinen et al. (2001) showed that fertilization caused increase of branch size and frequency.

Most of the variation between fertilized and nonfertilized trees could be explained by growth rate. The combination of higher MFA and decrease in density means that we can expect lower stiffness and strength of the wood from fertilized trees. An over 100% increase in ring width only corresponded to approximately a 20% decrease in wood density and the production of wood dry matter was hence increased by treatments.

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