Alternative chlorine-free peroxide for silicone rubber: Benefits with Aktisil Q

Author: Nicole Holzmayr

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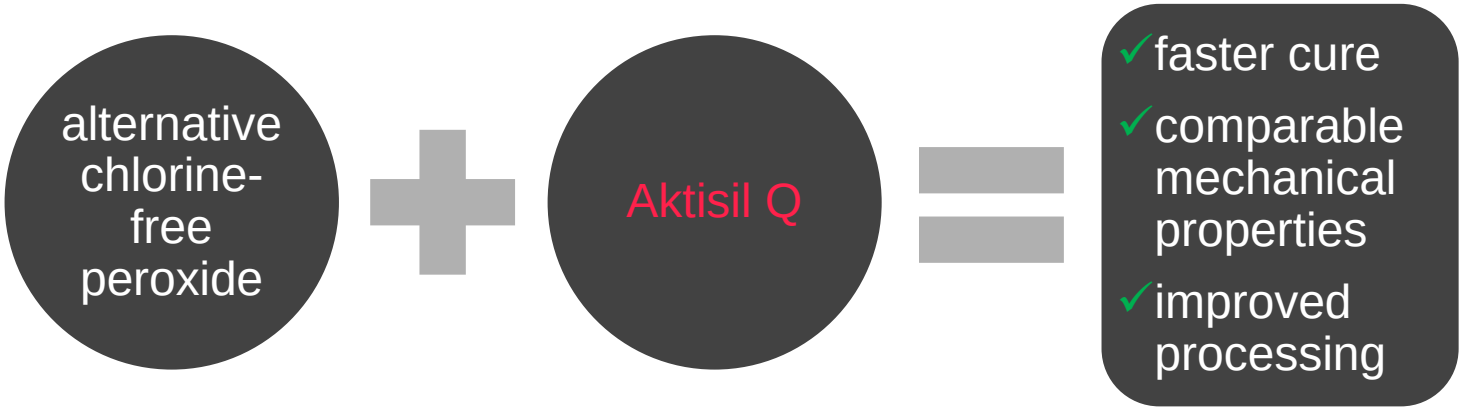
Status Quo

use of chlorinated peroxides	chlorinated peroxides	alternative chlorine-free peroxides
in solid silicone rubber mainly for extrusions	problematic due to emission of PCB*	✘ slower curing ✘ difficult processing (high stickiness of the compound during mixing and demolding)

* PCB: polychlorinated biphenyls

Objective



Formulation

		phr	
Elastosil R 401/40	polymer hardness: 40 Shore A	100	
Aktisil Q	filler Neuburg Siliceous Earth, methacrylic functionalized	0 – 100	
Perkadox PD-50S-ps DCBP	peroxide, chlorinated di-(2,4-dichlorobenzoyl)peroxide	1.5	-
Perkadox PM-50S-ps DMBP	peroxide, chlorine-free di-(4-methylbenzoyl)peroxide	-	1.07

Experimental Plan and Specimen Preparation

original conditions → head-to-head comparison → adjusted conditions

DCBP

cure temperature: 115 °C

DMBP

cure temperature: 115 °C

DMBP

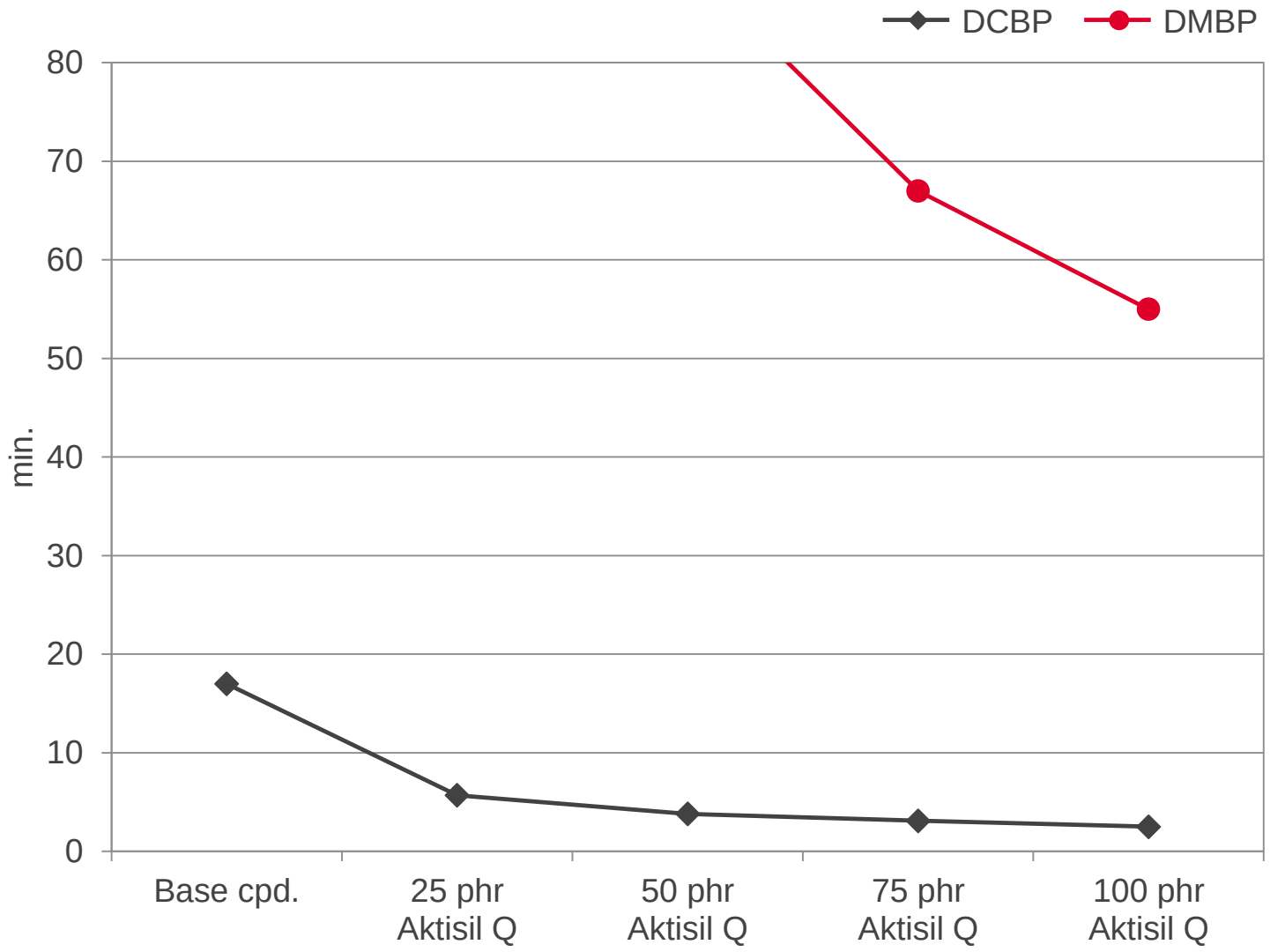
cure temperature: 135 °C

specimen preparation	press-cure	post-cure
duration	5 min.	4 h
temperature	as indicated	200 °C

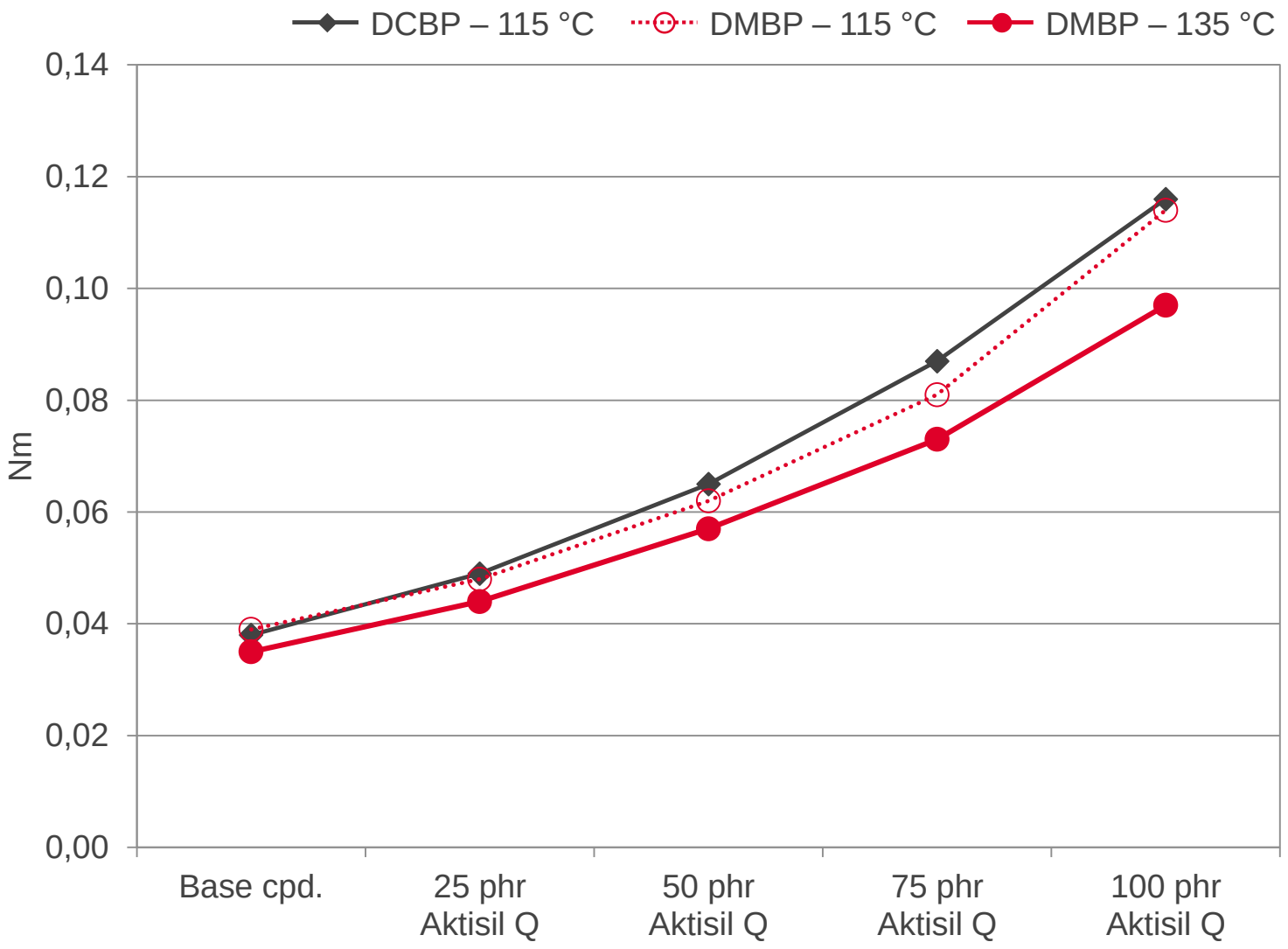
Processing

Base cpd. + DCBP	Base cpd. + DMBP	Base cpd. + DMBP + Aktisil Q
✓ no stickiness during mixing or demolding	✗ sticks during mixing and demolding	✓ no stickiness during mixing or demolding

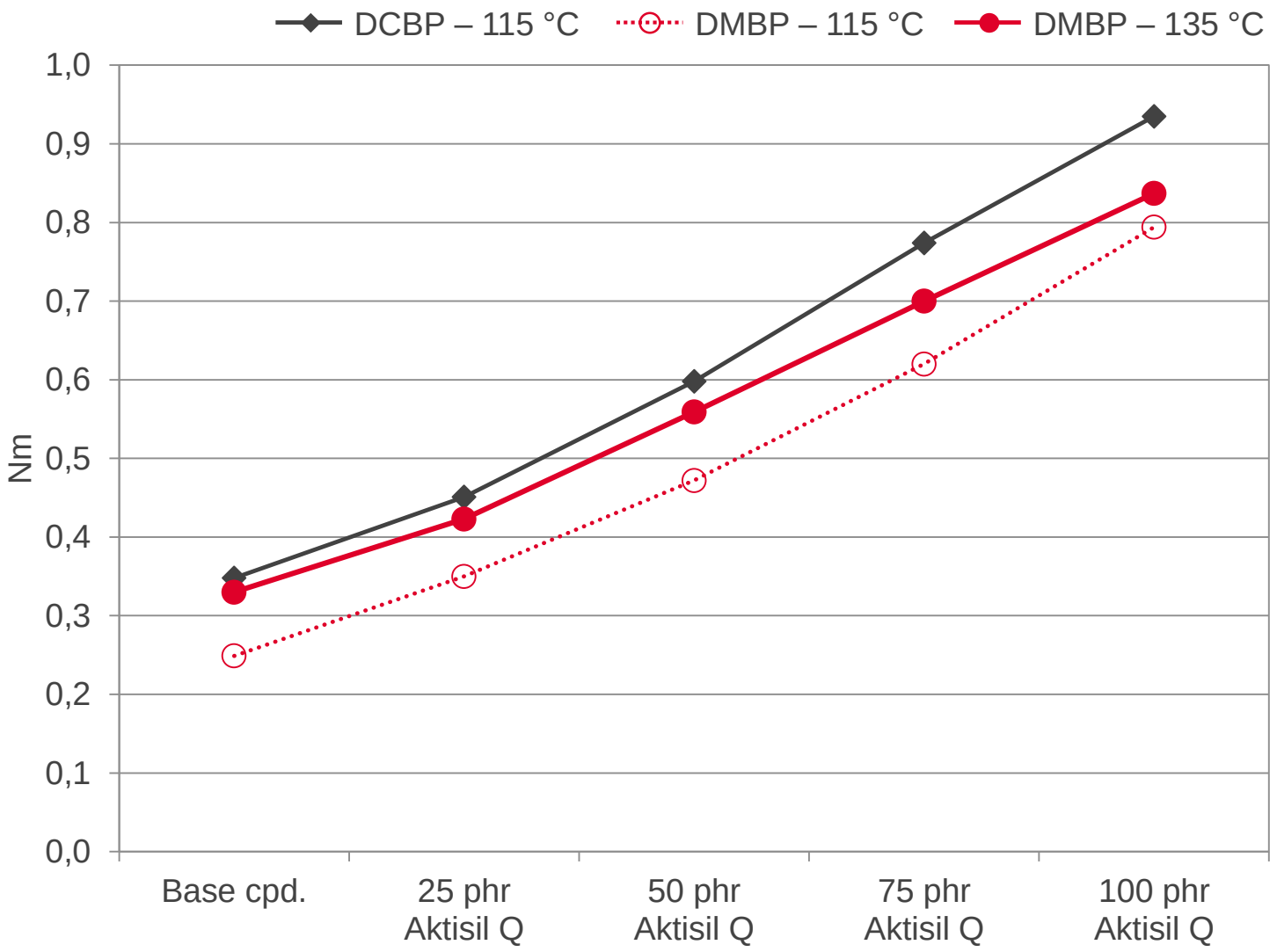
Scorch Behavior at 70 °C



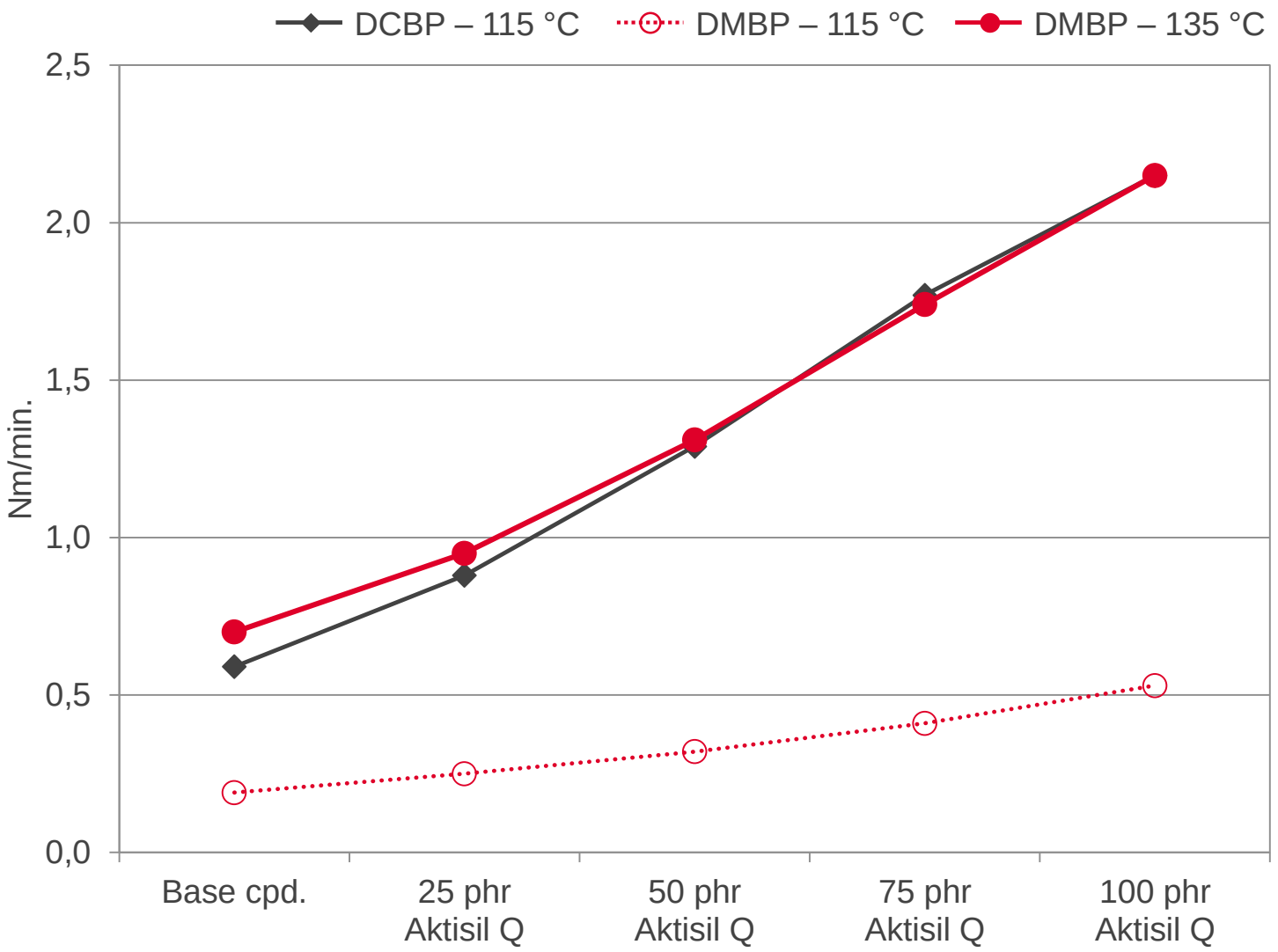
Torque Minimum



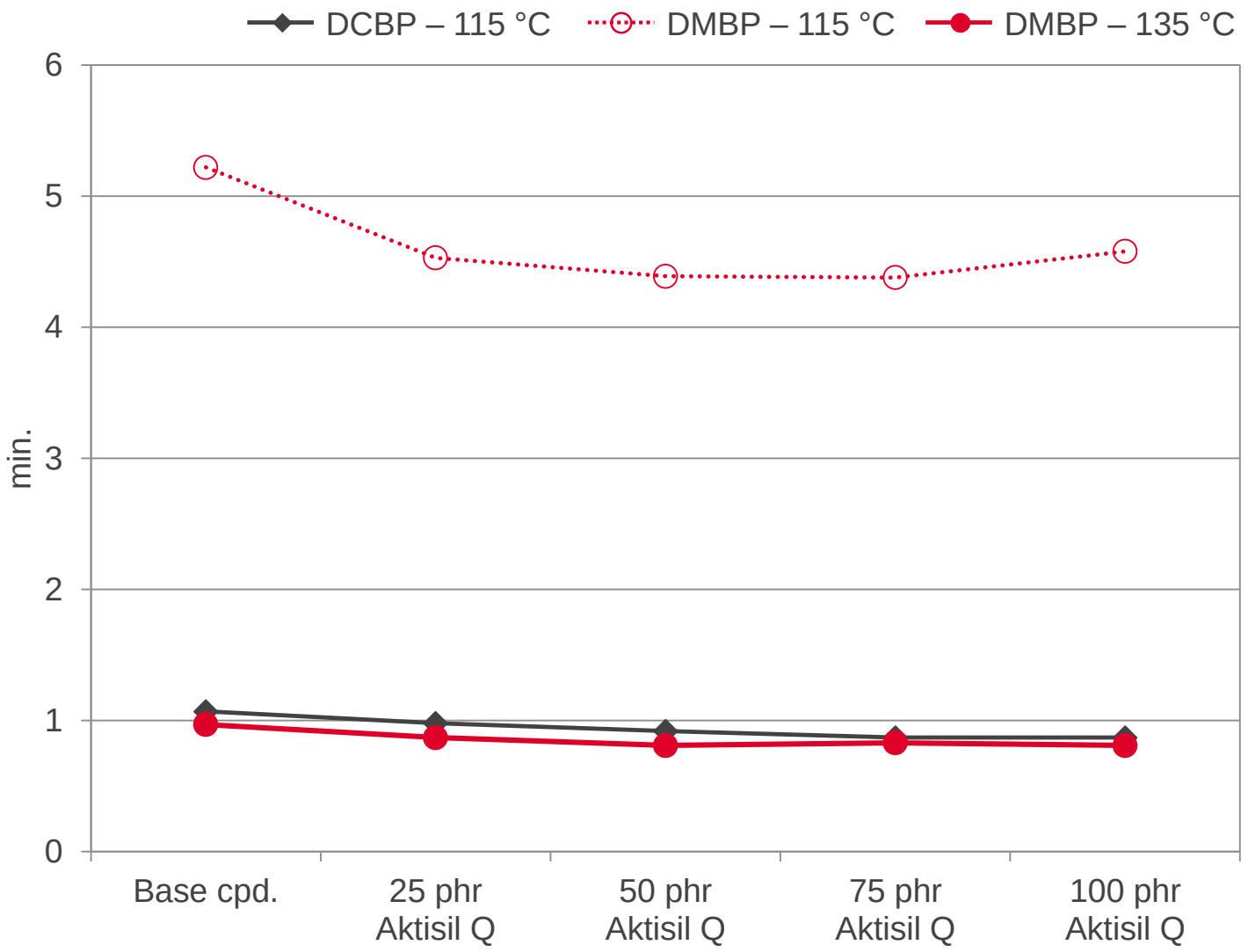
Cure Yield



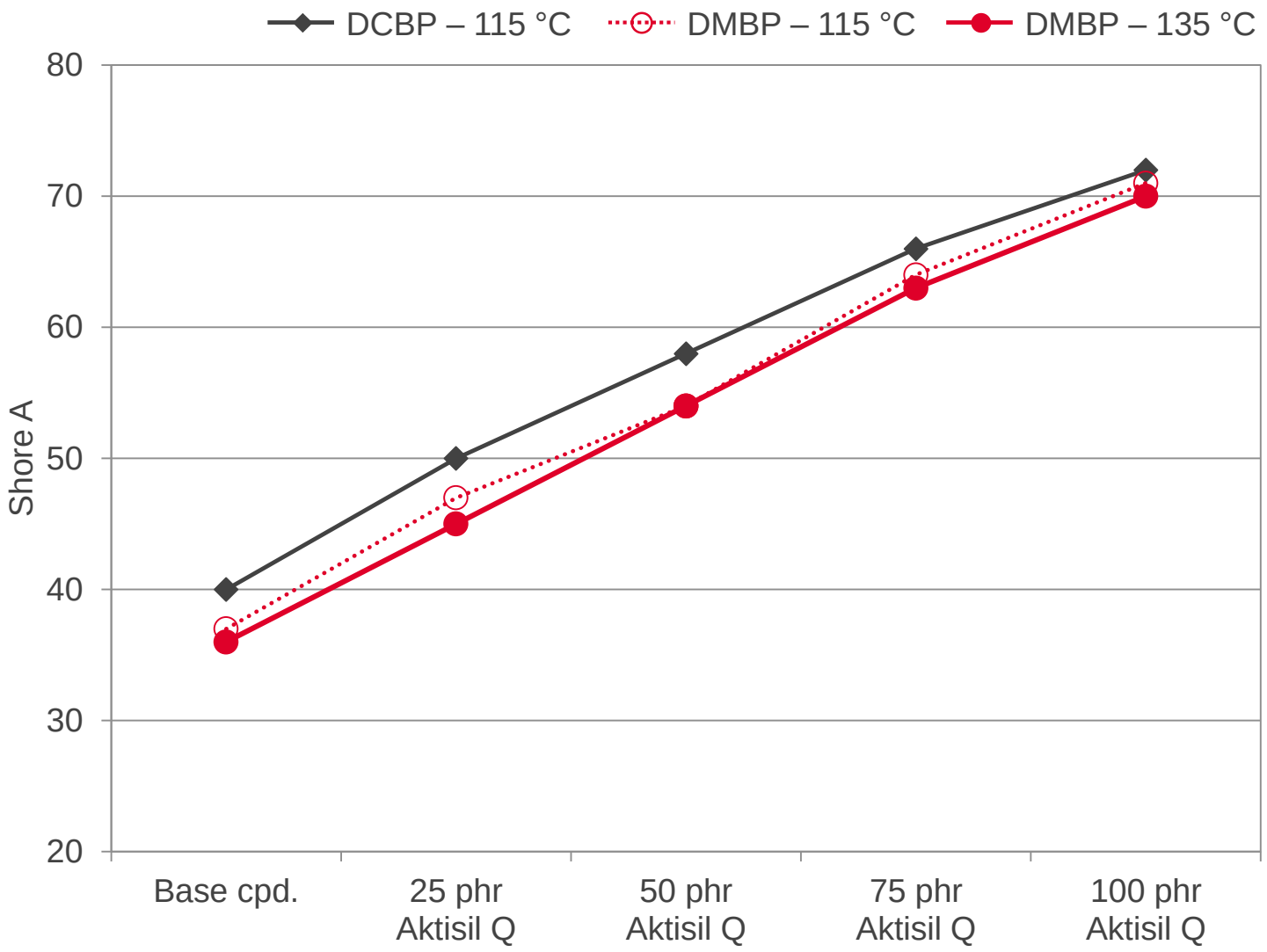
Maximum Cure Rate



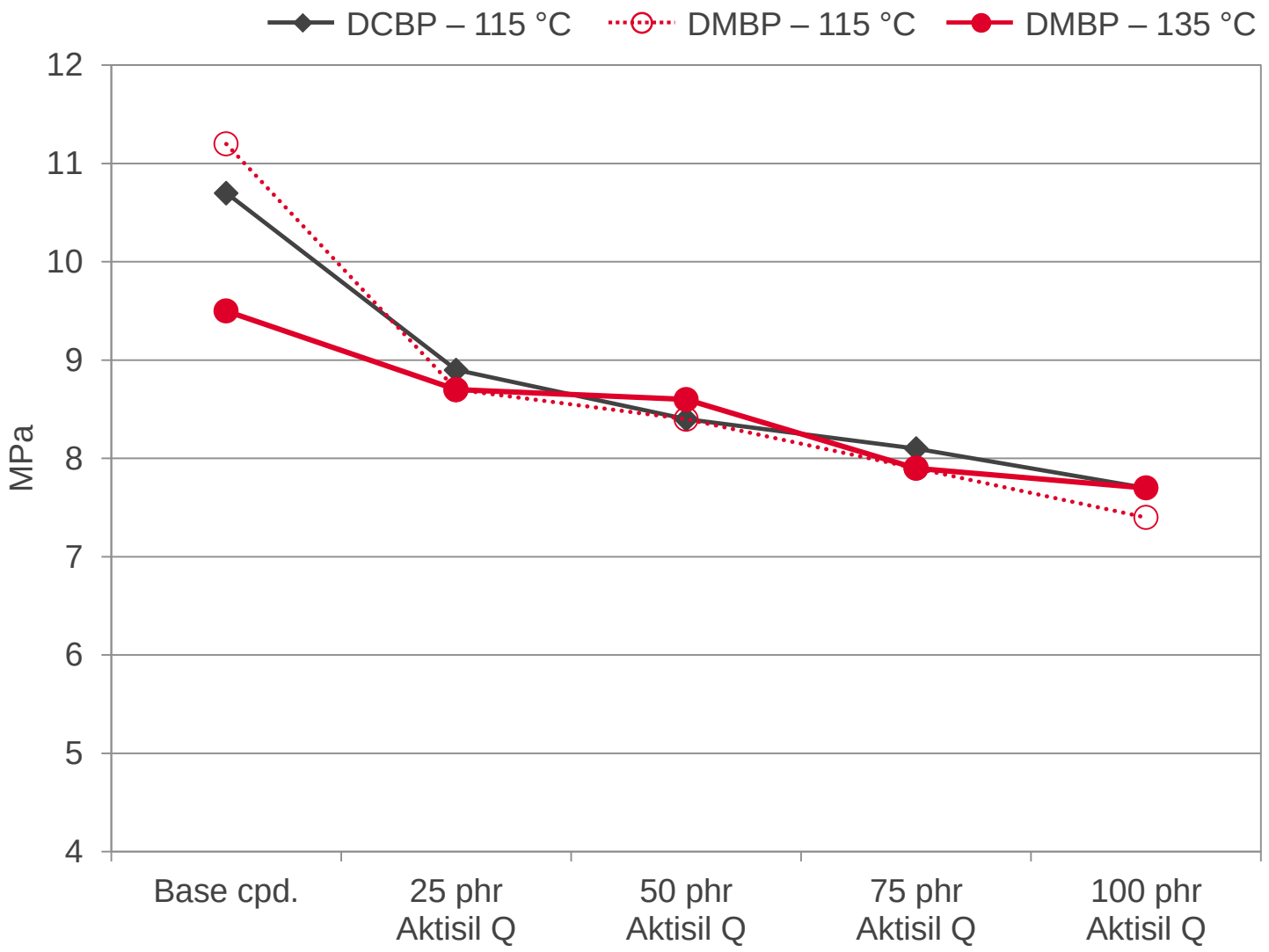
Cure Time t_{90}



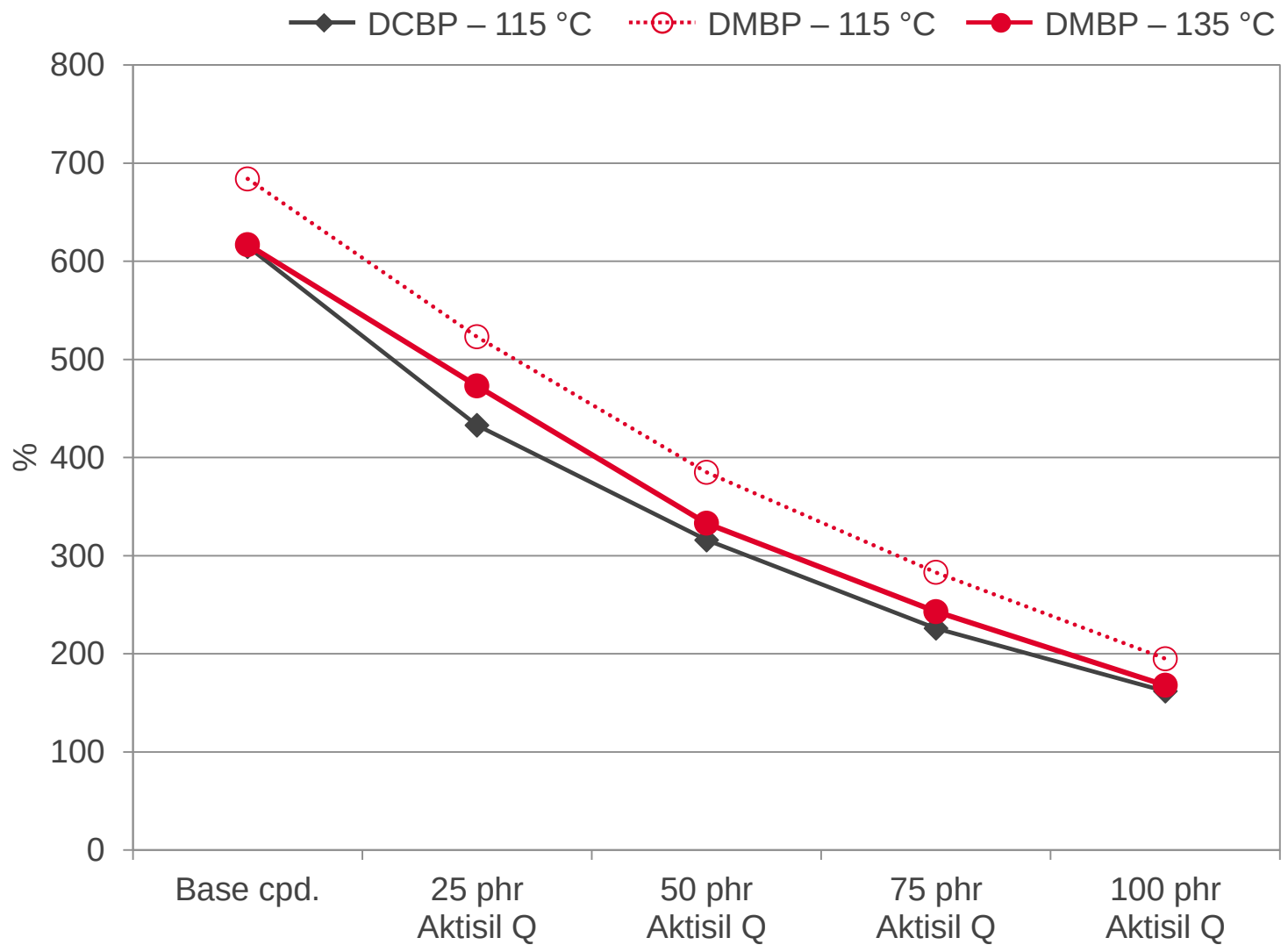
Hardness



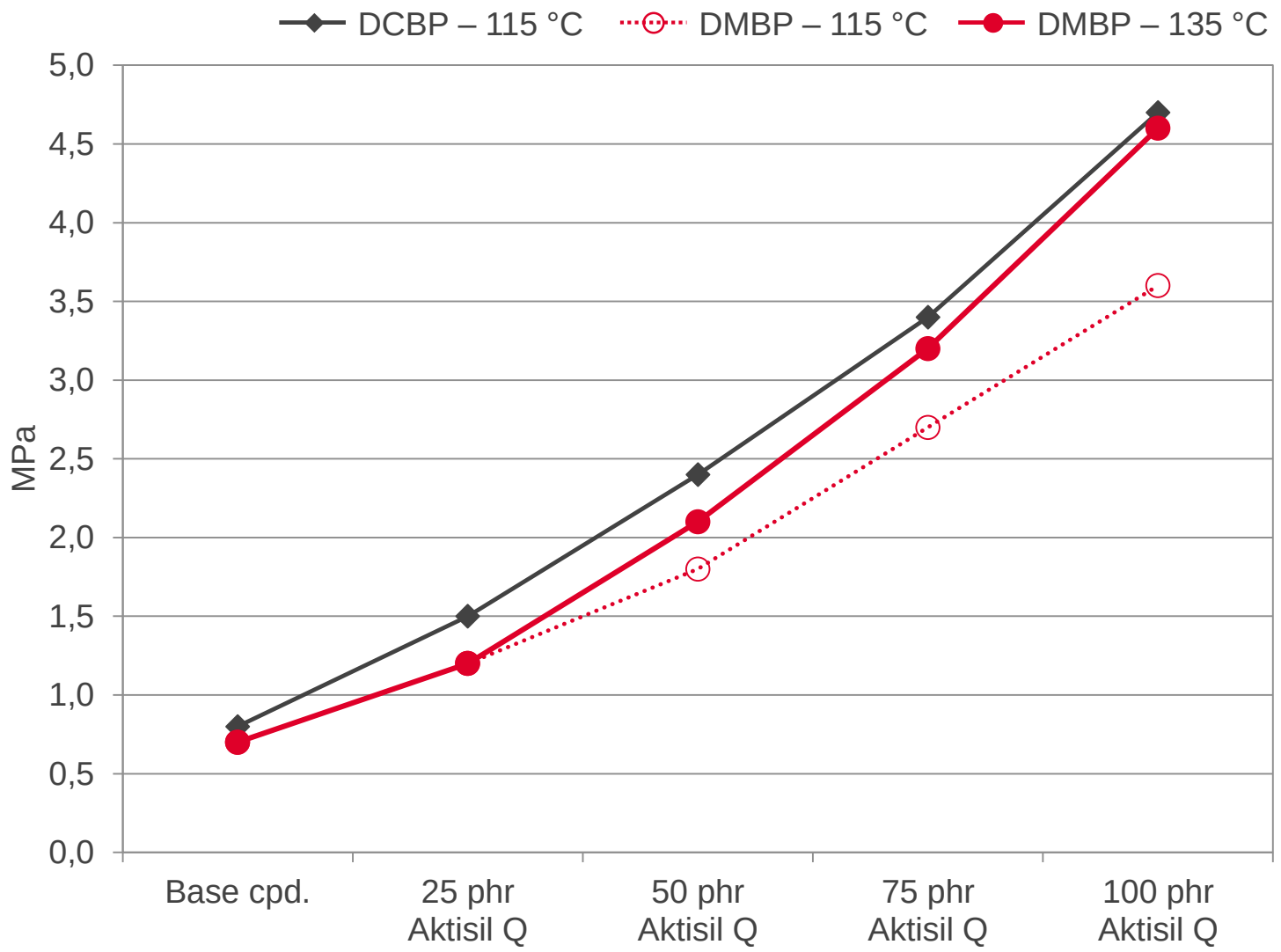
Tensile Strength



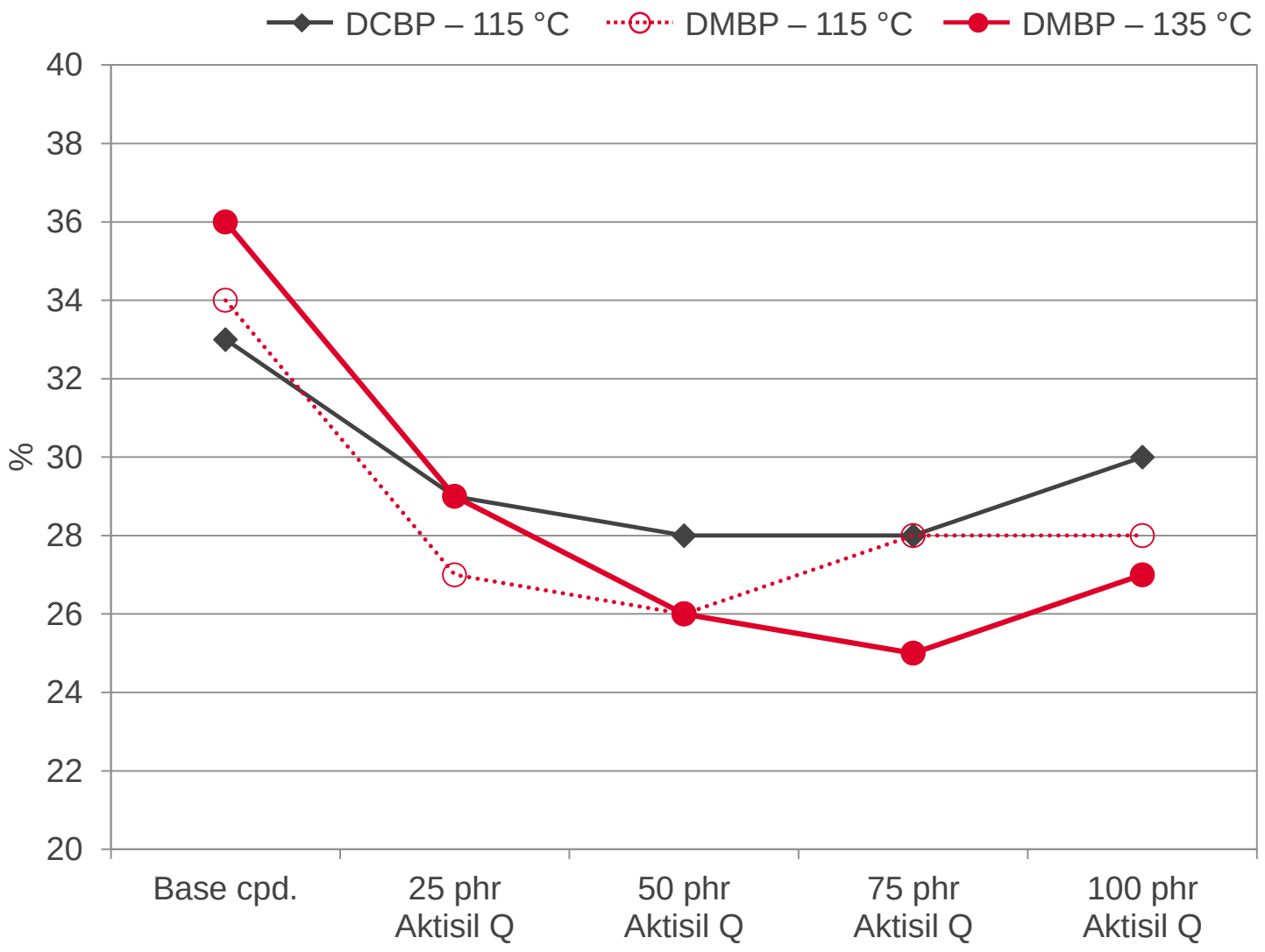
Elongation at Break



Modulus 100 %



Compression Set
24 h / 175 °C, 25 % defl.



Conclusion

- Replacing chlorinated peroxide with a chlorine-free alternative
- along with using Aktisil Q and increasing the cure temperature
- ✓
  - easy processing due to eliminated stickiness
  - higher scorch safety
  - comparable and fast curing
  - comparable tensile properties
  - slightly improved compression set

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HOFFMANN MINERAL GmbH
Muenchener Straße 75
DE-86633 Neuburg (Donau)

Phone: +49 8431 53-0
Internet: www.hoffmann-mineral.de
E-mail: info@hoffmann-mineral.com

Test Standards

Test	Standard
Mooney viscosity, ML 1+4	DIN ISO 289-1
Mooney scorch, ML +5	DIN ISO 289-2
Rotorless curemeter	DIN 53 529 Part 3
Hardness	DIN ISO 7619-1
Tensile strength	DIN 53 504, S2
Modulus 100 %	DIN 53 504, S2
Elongation at break	DIN 53 504, S2
Compression set	DIN ISO 815-1, B

Table of Results
DCBP (Perkadox PD-50S-ps), 115 °C

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		Base cpd.	25 phr Aktisil Q	50 phr Aktisil Q	75 phr Aktisil Q	100 phr Aktisil Q
Rheology						
Mooney viscosity, ML 1+2, 70 °C	MU	16	19	24	33	58
Mooney scorch, ML +5, 70 °C	min.	17	5.7	3.8	3.1	2.5
Rotorless curemeter M_{min}, 115 °C	Nm	0.04	0.05	0.07	0.09	0.12
Rotorless curemeter M_{max}-M_{min}, 115 °C	Nm	0.35	0.45	0.60	0.77	0.94
Rotorless curemeter V_{max}, 115 °C	Nm/min.	0.59	0.88	1.29	1.77	2.15
Rotorless curemeter t_{90}, 115 °C	min.	1.07	0.98	0.92	0.87	0.87
Mechanical properties – press-cure 5 min. / 115 °C; post-cure 4 h / 200 °C						
Hardness	Sh. A	40	50	58	66	72
Tensile strength	MPa	11	8.9	8.4	8.1	7.7
Modulus 100 %	MPa	0.8	1.5	2.4	3.4	4.7
Elongation at break	%	615	433	316	226	162
Tear resistance Trouser tear	N/mm	5.7	1.9	2.5	1.8	1.5
Tear resistance Graves	N/mm	22	9.3	7	6.4	6.1
Compression Set, 24 h / 175 °C, 25 % defl.	%	33	29	28	28	30

Table of Results

DMBP (Perkadox PM-50S-ps), 115 °C

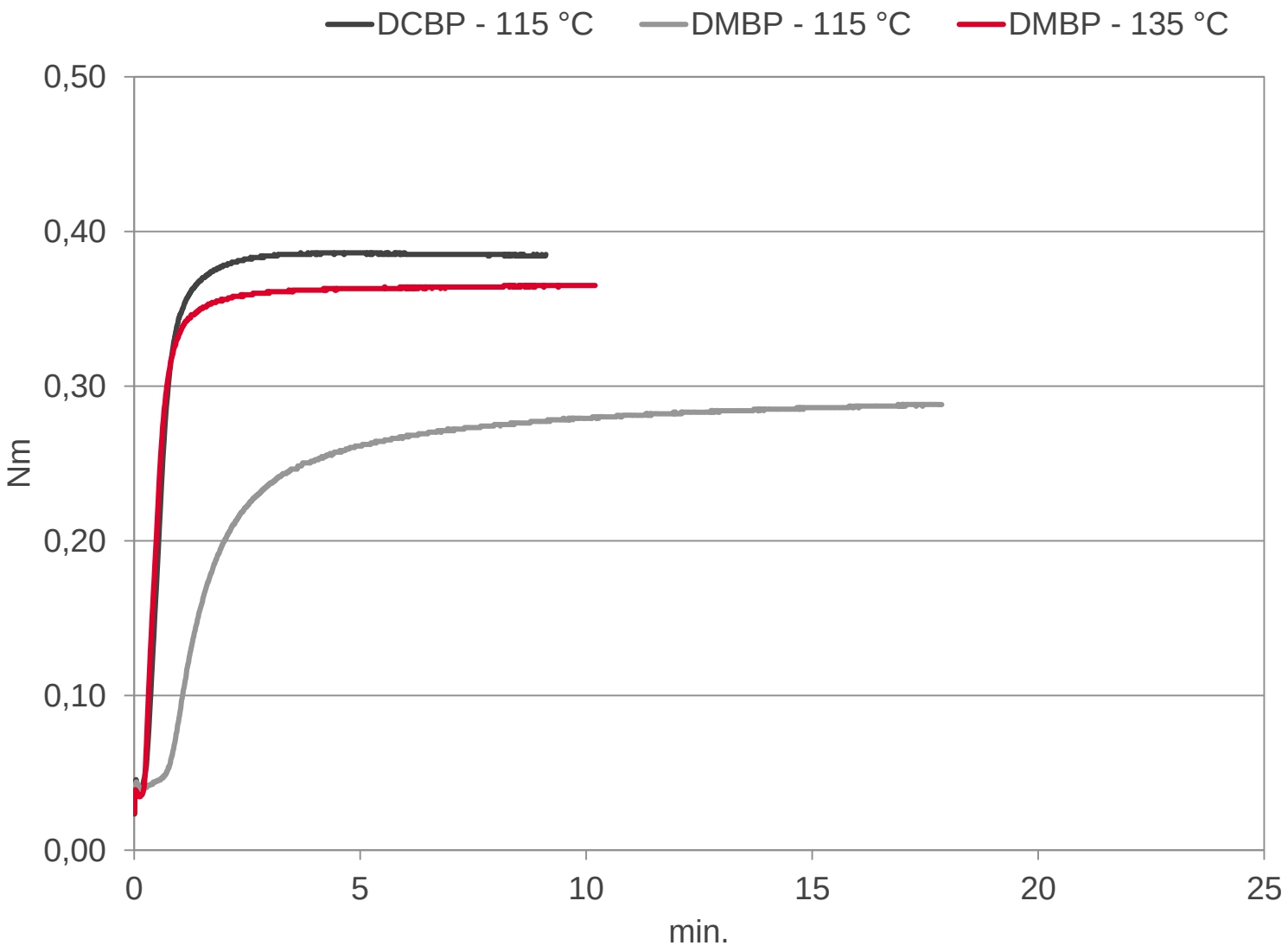
		Base cpd.	25 phr Aktisil Q	50 phr Aktisil Q	75 phr Aktisil Q	100 phr Aktisil Q
Rheology						
Mooney viscosity, ML 1+2, 70 °C	MU	16	19	23	27	36
Mooney scorch, ML +5, 70 °C	min.	> 90	> 90	> 90	67	55
Rotorless curemeter M_{min}, 115 °C	Nm	0.04	0.05	0.06	0.08	0.11
Rotorless curemeter M_{max}-M_{min}, 115 °C	Nm	0.25	0.35	0.47	0.62	0.80
Rotorless curemeter V_{max}, 115 °C	Nm/min.	0.19	0.25	0.32	0.41	0.53
Rotorless curemeter t_{90}, 115 °C	min.	5.2	4.5	4.4	4.4	4.6
Mechanical properties – press-cure 5 min. / 115 °C; post-cure 4 h / 200 °C						
Hardness	Sh. A	37	47	54	64	71
Tensile strength	MPa	11	8.7	8.4	7.9	7.4
Modulus 100 %	MPa	0.7	1.2	1.8	2.7	3.6
Elongation at break	%	684	523	385	283	195
Tear resistance Trouser tear	N/mm	7.0	7.3	3.6	2.3	1.8
Tear resistance Graves	N/mm	38	9.3	8.4	7.2	6.5
Compression Set, 24 h / 175 °C, 25 % defl.	%	34	27	26	28	28

Table of Results

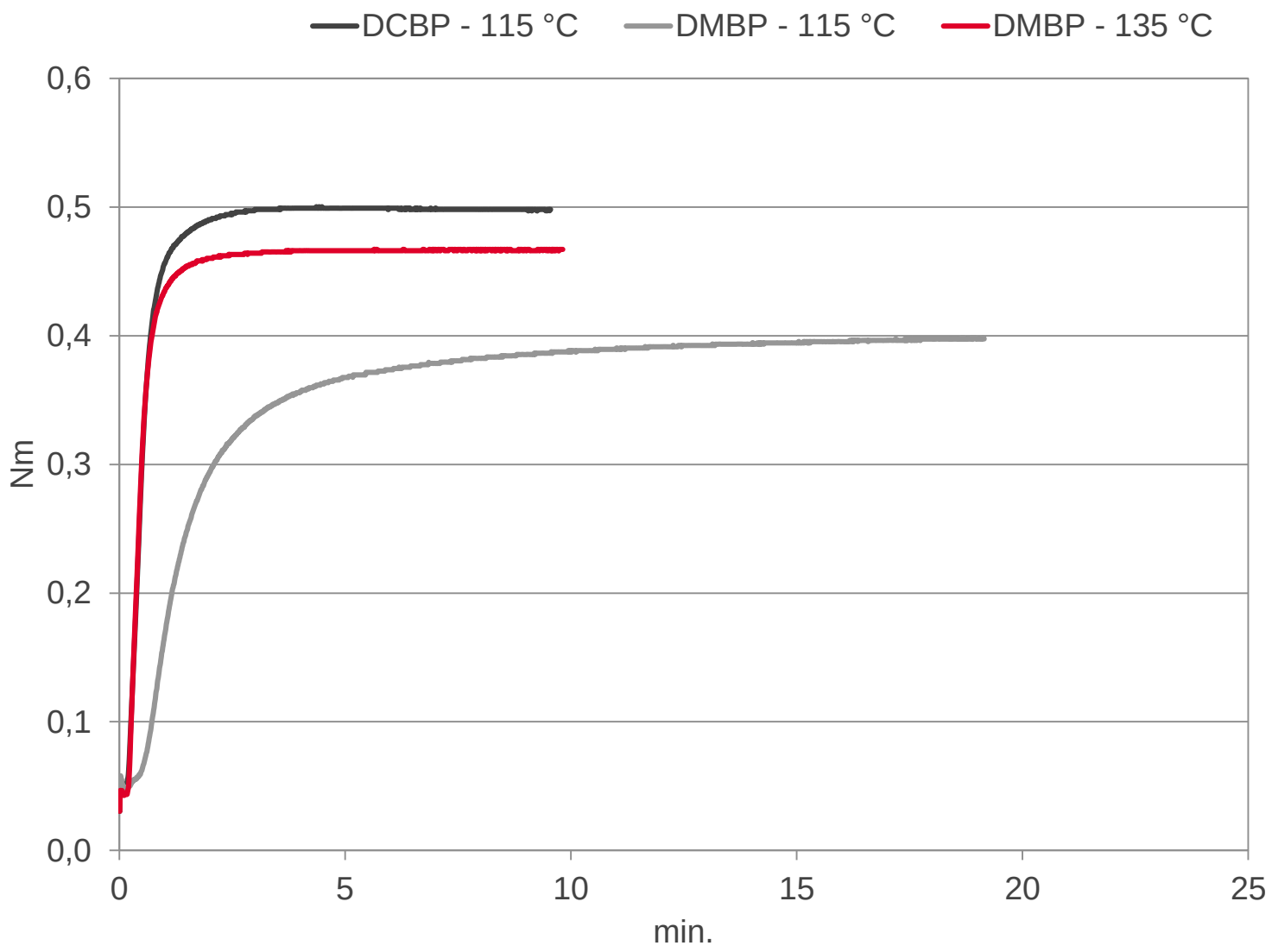
DMBP (Perkadox PM-50S-ps), 135 °C

		Base cpd.	25 phr Aktisil Q	50 phr Aktisil Q	75 phr Aktisil Q	100 phr Aktisil Q
Rheology						
Rotorless curemeter M_{min}, 135 °C	Nm	0.04	0.04	0.06	0.07	0.10
Rotorless curemeter M_{max}-M_{min}, 135 °C	Nm	0.33	0.42	0.56	0.70	0.84
Rotorless curemeter V_{max}, 135 °C	Nm/min.	0.70	0.95	1.31	1.74	2.15
Rotorless curemeter t_{90}, 135 °C	min.	0.97	0.87	0.81	0.83	0.81
Mechanical properties – press-cure 5 min. / 135 °C; post-cure 4 h / 200 °C						
Hardness	Sh. A	36	45	54	63	70
Tensile strength	MPa	9.5	8.7	8.6	7.9	7.7
Modulus 100 %	MPa	0.7	1.2	2.1	3.2	4.6
Elongation at break	%	617	473	333	243	168
Tear resistance Trouser tear	N/mm	7.0	4.9	5.1	2.3	1.7
Tear resistance Graves	N/mm	35	10	8.1	7.1	6.4
Compression Set, 24 h / 175 °C, 25 % defl.	%	36	29	26	25	27

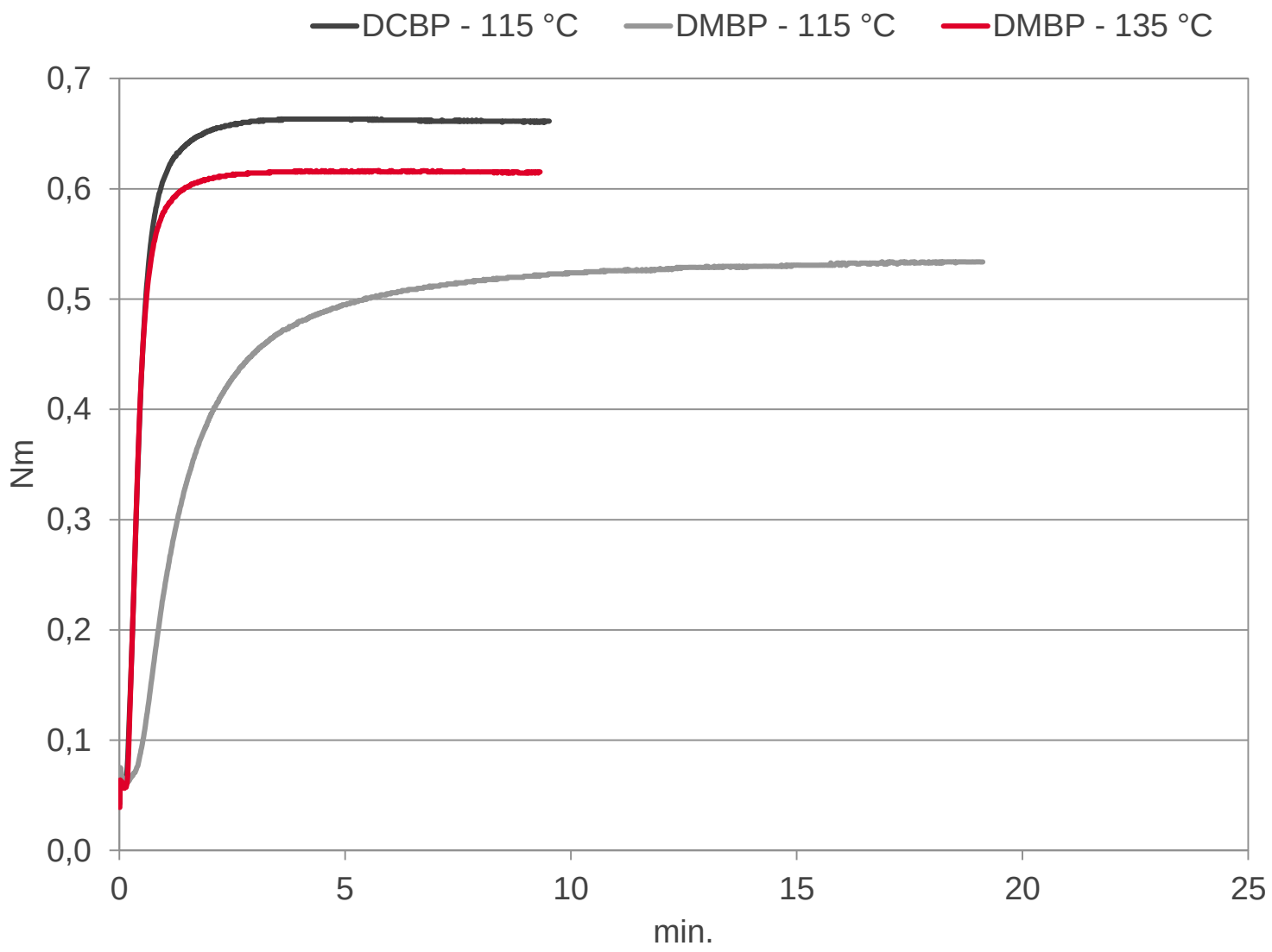
Cure Properties
Base compound



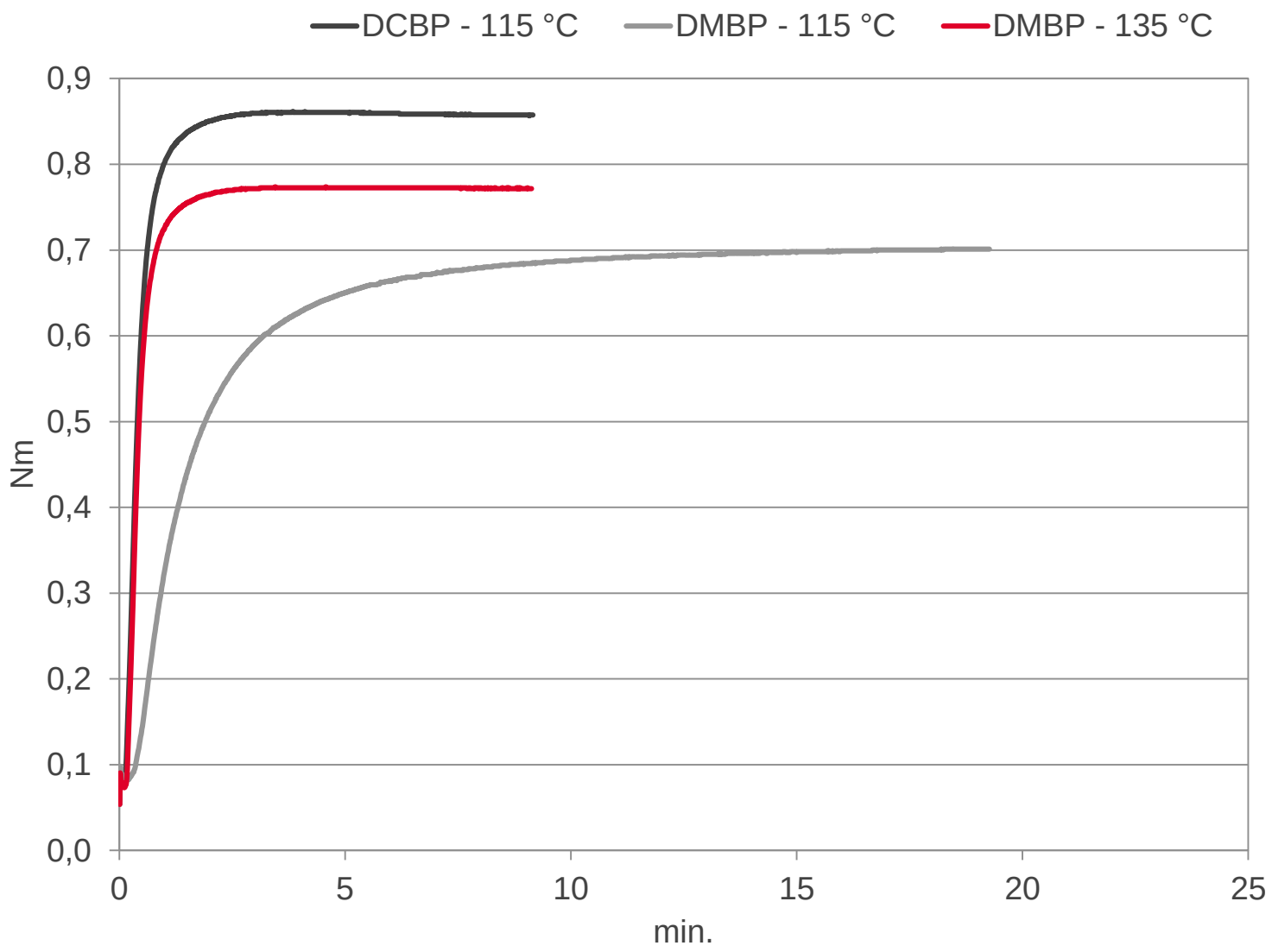
Cure Properties
25 phr Aktisil Q



Cure Properties
50 phr Aktisil Q



Cure Properties
75 phr Aktisil Q



Cure Properties
100 phr Aktisil Q