

Bulab[®] 600HP

Previous in-house testing showed that adding Bulab 600HP to solvent-based epoxy coatings decreased cure time. Recent advances in analytical methods allowed for the testing of not only cure rate as a function of Bulab 600HP concentration, but also cure rate as a function of concentration and temperature.

The base coating for these tests is displayed in the table below. The epoxy/amine ratio was adjusted to a theoretical 1:1. Bulab 600HP was post added to Part A of the formulation and the concentration range was evaluated from 0–5% at 25°C, and from 0–3% for all other temperatures studied.

B36P292A Epoxy Formulation – Part A			
Item	Target weight (grams)	Actual weight (grams)	Calculated Epoxy Equivalents (Part A)
Charge the following under agitation to PT1 vessel equipped with 70 mm cowles blade			
EPOTUF [®] 38-505	354.10	354.10	
DOWANOL [™] PM	29.20	29.23	
DOWANOL [™] DPM solvent	29.20	29.20	
CYMEL [®] U-21-511	12.70	12.69	
Anti-Terra [®] -U	2.70	2.72	
Methyl isobutyl ketone (MIBK)	36.80	36.82	
Mix 15 min @ 630 rpm; add the following under agitation			
MFP	204.60	204.60	
R5098D Copperas [®] Red Iron Oxide (Dark)	153.50	153.50	
BENWOOD [™] TALC 2207	108.00	108.00	
WG [™] 325 muscovite mica	35.60	35.60	
#1 Barytes BB-2501	138.90	138.90	
Hock @ 5480 rpm (20 m/s) – 4 min, then 4300 rpm 3 min, followed by 3 min @ 3390 rpm			
Mix 10 min @ 620 rpm – Stop, raise blade, scrape sides and bottom			
Hock @ 5480 rpm (20 m/s) – 4 min, then 4300 rpm 3 min, followed by 3 min @ 3390 rpm			
Check Grind – Dirty 5.5 N.S.			
Total Part A	1105.30	1105.36	0.5058571

B36P292B Epoxy Formulation Crosslinker – Part B			
Item	Target weight (grams)	Actual weight (grams)	Calculated Amine Equivalents (Part B)
EPOTUF 37-650	65.76	65.76	
Xylene	0.70	0.70	
Total Part B	66.46	66.46	0.505856

Total Formulation	1171.76
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Epoxy cure as a function of Bulab® 600HP addition

Initial testing showed that at 25°C gel time (indicated by the crossover point, Figure 1) and higher viscosity (Figure 2) was reached in a shorter amount of time as Bulab 600HP concentration increased.

Figure 1. Loss/storage modulus curves showing gel points at 25°C

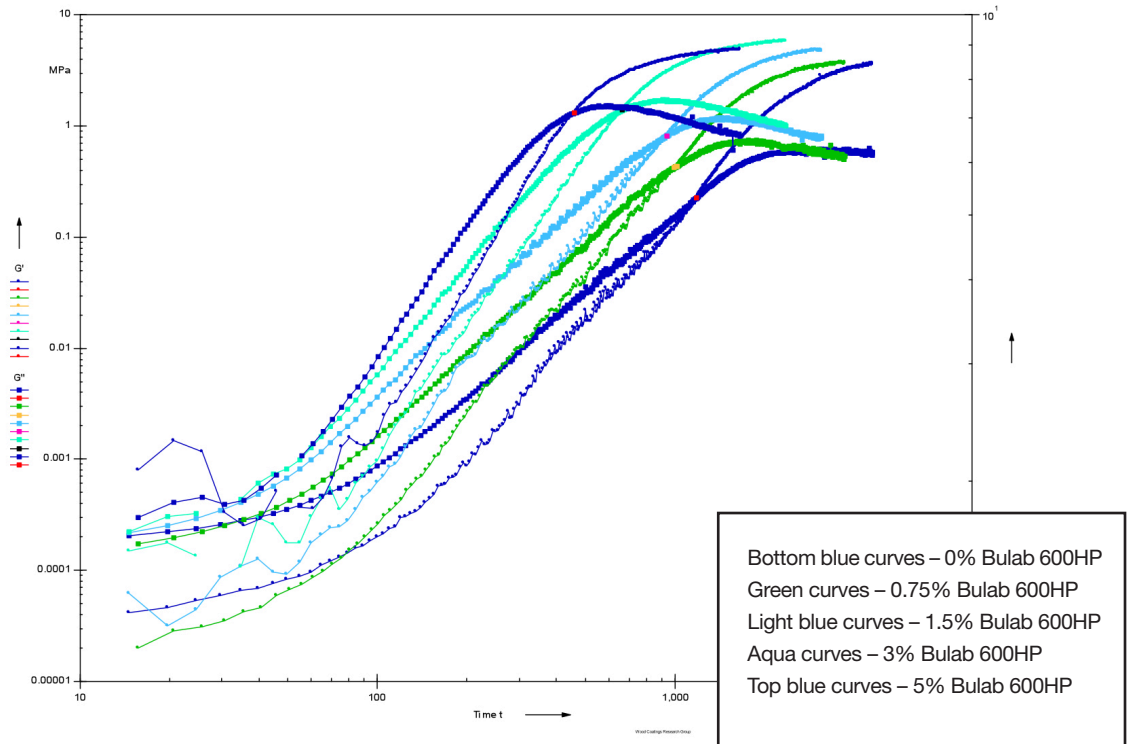
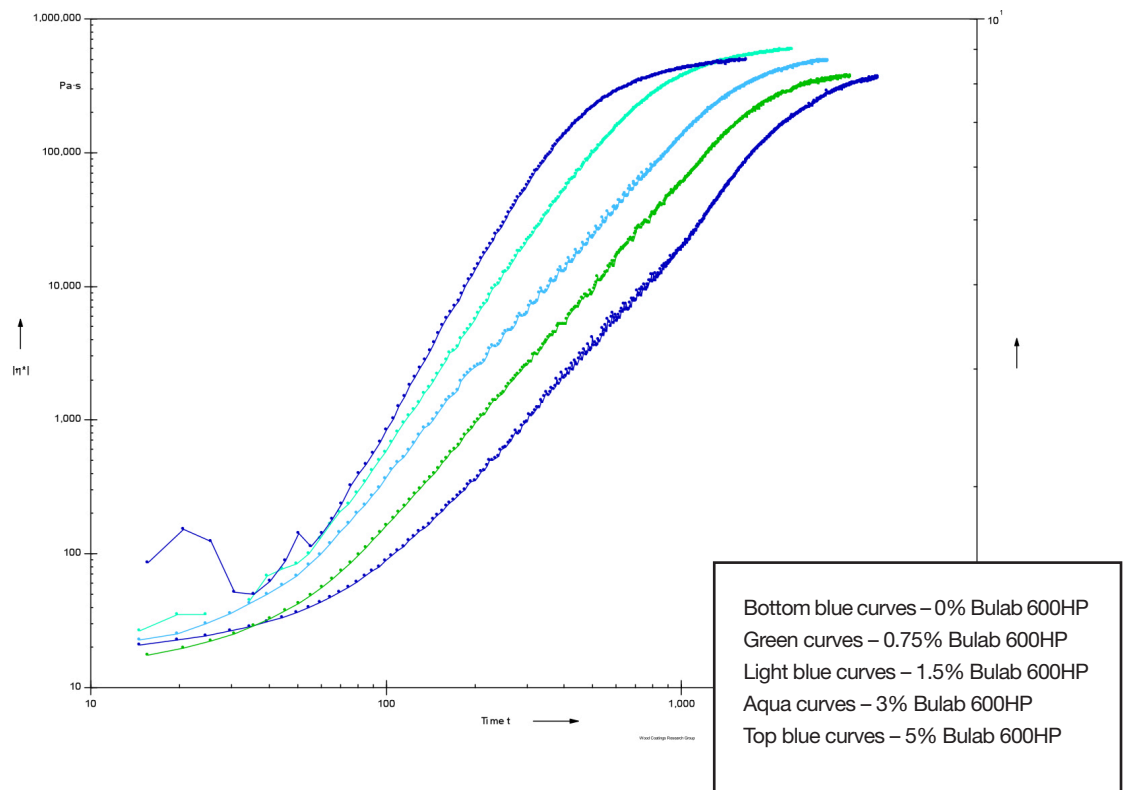


Figure 2. Complex viscosity as function of Bulab 600HP at 25°C



Epoxy cure as a function of Bulab® 600HP addition and temperature

The study next determined that Bulab 600HP increased the rate of solidification and crosslinking of the epoxy coating studied at each temperature investigated. Curing rate increased as the concentration of Bulab 600HP increased, with the exception of an aberration observed at 0.75% at 10°C and 4.4°C relative to 0% Bulab 600HP (Figures 3 and 4). As temperatures decrease from 25°C, the impact of Bulab 600HP becomes greater, resulting in a reduced gel time relative to 0% Bulab 600HP. The 15.5°C curve shows the greatest slope while the 25°C curve the smallest slope (Figures 3 and 4).

Figure 3. Impact of temperature on gel time (G'/G'' crossover) as a function of Bulab 600HP concentration

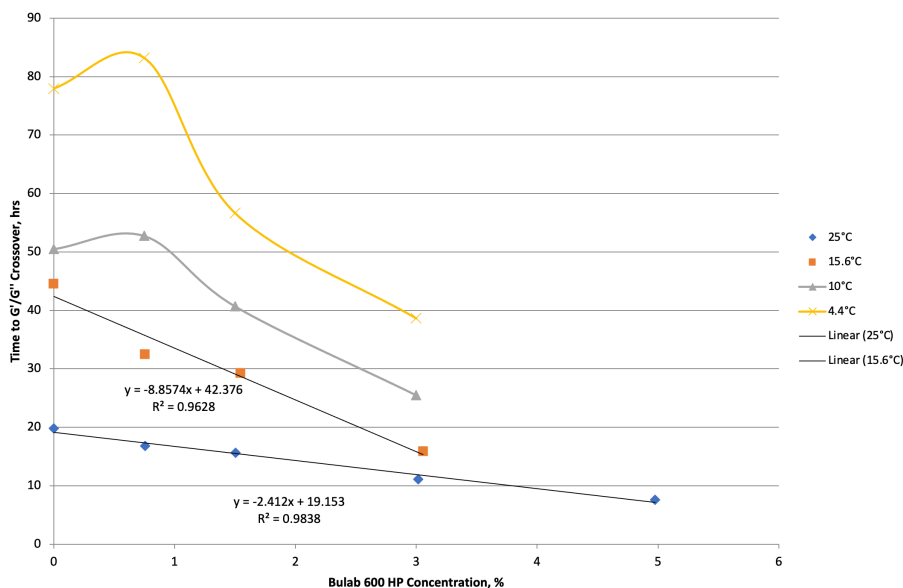


Figure 4. Impact of temperature on linear gel time (G'/G'' crossover) as a function of Bulab 600HP concentration

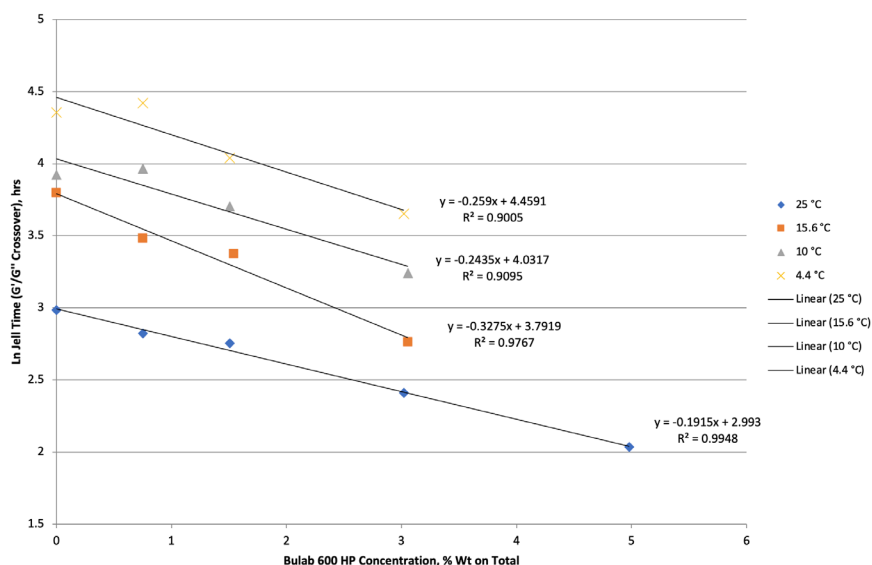


Figure 5 is an Arrhenius plot analyzing the effect of gel time as a function of temperature at a constant Bulab® 600HP concentration. It is clear from the data that adding Bulab 600HP dramatically improves cure of the epoxy vs. 0% Bulab 600HP, with 0.75% Bulab 600HP showing the greatest slope for the concentrations studied.

Correlation coefficients (R^2 values) are such that one may confidently predict cure-time as a function of Bulab 600HP concentration and cure temperature.

Figure 5. Linear gel time vs temperature at constant Bulab 600HP concentration

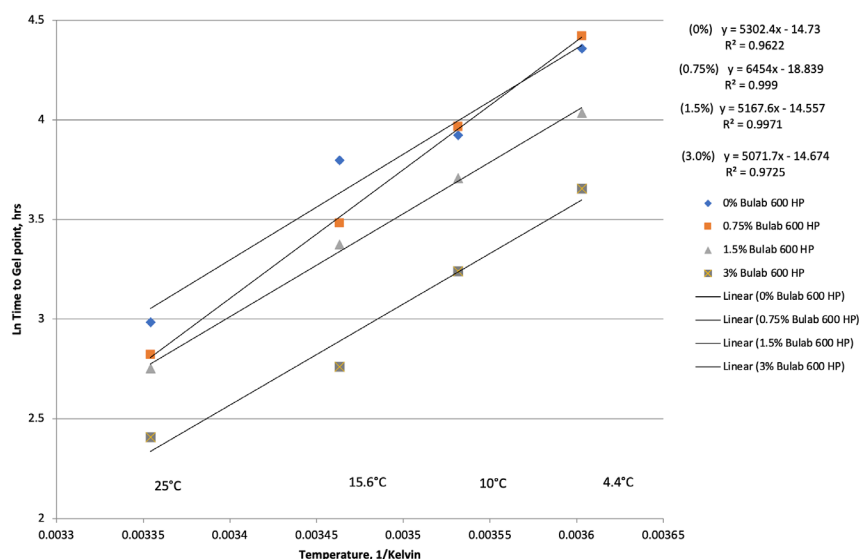
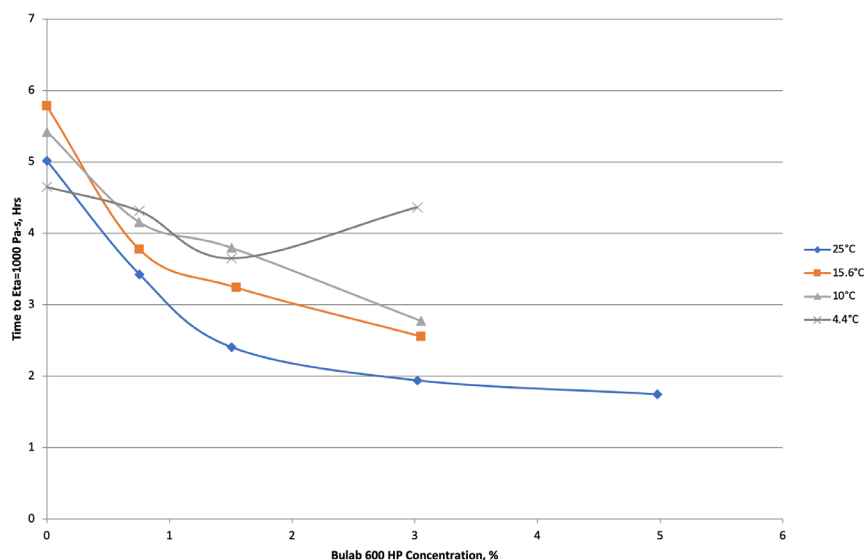


Figure 6 displays the time for the films to reach a complex viscosity of 1000 Pa·s as a function of Bulab 600HP concentration and temperature. Except for the 3% Bulab concentration at 4.4°C, the time to reach 1000 Pa·s decreased with increasing Bulab 600HP concentration at each temperature tested.

Figure 6. Impact of temperature on time $\eta=1000$ Pa·s as a function of Bulab 600HP concentration



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