Taking the next step in fast curing 2K NCO technology

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Abstract
Obtaining an improved balance between drying time and pot life is an ever-ongoing challenge in 2K isocyanate coatings. In this paper, allnex presents technology that takes 2K NCO curing to the next level, shortening the drying time down to 1 hour at room temperature, while increasing the pot life. In addition, the open time can be tuned and early hardness and chemical resistance are significantly improved.

Keywords: 2K isocyanate, fast curing, pot life-drying balance.

Introduction
In 2K isocyanate curing coatings, there is an ever-ongoing search for faster drying and drying at lower temperatures, while retaining or even improving the pot life. Over the past years, allnex has introduced a number of new technologies, bringing drying and handling times down from originally 6-8 hours for high solids, VOC-compliant systems, to 2.5 hours at room temperature, while still having very good pot life and working time.

Now, allnex has taken the next step and has introduced a new product range based on new Fast Cure (FC) technology, decreasing the drying down to 1 hour at room temperature. Examples in this paper focus on the application of the SETALUX® FC acrylic polyol range in clear coats for vehicle refinishes and on the application of the DUROFTAL® FC polyester polyol range in pigmented top coats for general industrial and DTM applications, but this technology is also applicable for other markets, such as ACE, marine & protective, OEM plastics, wood and flooring.

Clear coat application performance
Clear coat formulations were prepared using the new FC technology and were compared to similar clear coat paints based on standard products. The formulations were applied by spraying on both inert substrates and substrates pre-coated with a waterborne basecoat. Application results for both medium and high solids grades clearly show that paints based on the new FC technology show much shorter drying times, with similar or even significantly increased pot life. Clear coats based on FC resins have higher early hardness: depending on the application, this will result in better early handleability, better early polishability or sandability, and improved early chemical resistance.

SETALUX® FC medium solids acrylic polyols
SETALUX® FC 1227 BA-67 is a medium solids acrylic polyol with 3.6% OH, delivered at 67% solids content in butyl acetate (BAC).

The resin was tested in a clear coat formulation with Tolonate HDT-90 hardener (NCO/OH = 1.1); Dibutyltin dilaurate (DBTL) catalyst level was optimised for best overall performance; and MODAFLOW® Lambda was used as flow agent for best appearance. The application results reported in Table 1 clearly demonstrate the advantages of the FC product with respect to a standard medium solids product (data in bold).

Table 1: Comparison of application data of new FC technology with standard medium solids system

	SETALUX® FC 1227 BA-67	SETALUX 1198 SS-70
Tack-free time (h:mm) @ RT	**1:00**	4:10
Working time (+ 4s DC4)	**2:20**	1:00
Pot life (double viscosity, h:mm)	**3:00**	2:05
Persoz / König hardness		
• after 1 day @23°C	**240/132**	168/75
• after 30 min @60°C + 1 h @23°C	**235/128**	138/56
• after 7 days @23°C	262/148	345/206
Xylene resistance		
• after 1 day @23°C	**++**	-
• after 7 days @23°C	++	++
Long wave[1]	6	4.4
Short wave[1]	4.3	9.0
Gloss at 20°	90	91
DOI	91	95
Adhesion on Basecoat	++	++

[1]A BYK-Gardner Wavescan instrument was used to measure orange peel or surface 'waviness' at wavelengths between 1 and 10 mm (Long wave) and between 0.3 and 1 mm (Short wave). Values are given between 0 for a completely smooth surface (mirror) and 100 for a very bad orange peel.

The plot in Figure 1 shows that SETALUX® FC 1227 BA-67 has the better pot life-drying balance and superior early hardness: the size of the circles correlates with the hardness measured 1 hour after curing for 30 minutes at 60°C. Even at 33°C, double viscosity pot life is 135mins and working time (+4 s DC 4) is 105mins (not shown in the plot), making this resin very suitable for applications with less climate control and/or in Mediterranean and tropical areas. In addition to the above application data related to application in vehicle refinishes, SETALUX® FC 1227 BA-67 is also particularly useful for application in clear coats for the wood market.

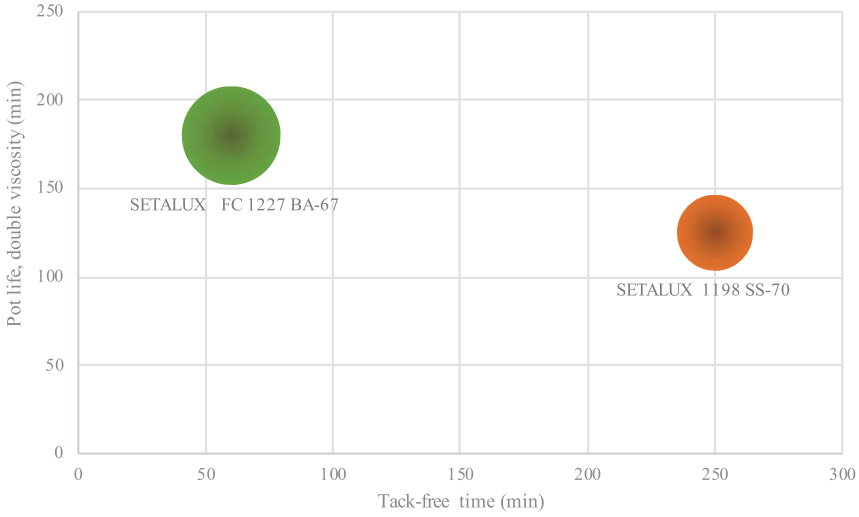

Figure 1: Pot life-drying balance comparison of new Fast Cure technology with a standard medium solids system: the size of the circles correlates with the hardness measured 1h after curing for 30 minutes at 60°C

SETALUX® FC high solids acrylic polyols.

SETALUX® FC high solids acrylic polyols are all delivered at 75% solids in butyl acetate. The main features of the different grades are described in Table 2.

The resins were tested in clear coat formulations with Tolonate HDT-LV hardener (NCO/OH = 1.2); DBTL catalyst level was optimised for each formulation for best overall performance; MODAFLOW® Lambda was used as flow agent for best appearance. VOC content for all formulations is below 420 g/L. Application results obtained with these three resins are summarised in Table 3 and compared with those of fast and standard high solids products.

Table 2: SETALUX® FC high solids grades

SETALUX® FC 1922 BA-75	4.1% OH	- High-end - Fastest RT drying - Excellent early hardness at RT - Very good pot life-drying balance
SETALUX® FC 1923 BA-75	3.6% OH	- Fast drying economy grade - Very long working time - Very good early hardness
SETALUX® FC 1925 BA-75	4.1% OH	- High performance economy grade - Ultrafast RT drying - Very good pot life-drying balance

Table 3: Comparison of application data of new FC technology with standard high solids systems

	Best in class **SETALUX® FC 1922 BA-75**	**Eco, 3.6% OH** **SETALUX® FC 1923 BA-75**	**High Perf. Eco, 4.1% OH** **SETALUX® FC 1925 BA-75**	**Fast** **SETALUX® 1915 BA-75**	**Standard** **SETALUX® 1910 BA-75**
DBTL (based on resin solids)	0.2	0.15	0.15	0.075	0.05
Dry film thickness (µm)	44	46	41	43	44
Tack-free time (h:mm) @ RT	**1:20**	2:10	**1:00**	2:40	4:55
Time to polish @ RT (after application)	**≤ 2:00**	~ 3:00	**~ 2:00**	~ 3:30	≤ 8:00
Working time (+4s DC 4) / Pot life (double viscosity)	1:00 / 1:15	**2:10 / 3:00**	1:30 / 1:50	< 0:20 / 1:20	1:00 / 2:30
Persoz hardness **(after tack-free)**:					
• 1 hour @23°C	**78**	33	**78**	28	35
• 4 hours @23°C	**159**	65	**112**	41	59
• 1 day @23°C	**239**	142	187	151	171
• 1 week @23°C	**257**	216	204	224	275
Xylene resistance					
• after 1 day @23°C	+	+	+	+/-	+/-
• after 7 days @23°C	++	++	++	++	++
Long wave / Short wave	4.2 / 5.9	2.7 / 1.1	1.5 / 1.5	6 / 1.6	1.8 / 0.4
Gloss at 20°	89	90	88	89	90
DOI	96	96	96	96	96

Pot life, tack-free time and early hardness of the SETALUX® FC polyols in comparison with reference data are also shown in the plot in Figure 2: the size of the circles correlates with the hardness measured 1 hour after the tack-free time at RT.

Data in Table 3 and Figure 2 show that the new FC products have in general faster drying combined with a longer pot life and higher early hardness. Especially important is the shorter polishability time observed. This will contribute to profitability of paint users, because objects can be handled much earlier, which reduces the object cycle time significantly.

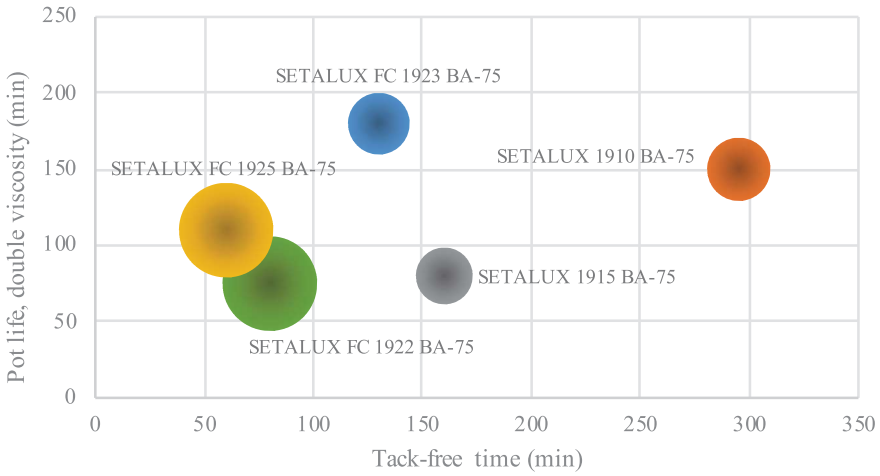

Figure 2: Pot life-drying balance comparison of new Fast Cure technology with standard high solids systems; the size of the circles correlates with the hardness measured 1 hour after tack-free time at RT

Study of clear coat conversion and coating properties

The improved performances of FC-based clear coats are explained by the higher conversion shortly after curing. This was demonstrated by FT-IR spectroscopy (Figure 3), showing that the new Fast Cure technology not only gives higher conversions, but also a longer open time, which can be finetuned by the choice of the optimal concentrations of tin catalyst and other formulation components (see formulation advice later). The longer open time also enables better levelling, leading to improved appearance.

The improvement of early chemical and mechanical properties was further supported by DMTA measurements, as shown in Table 4. These data prove that the ultimate properties are already achieved shortly after curing and that the glass transition temperature, as well as the crosslinking density, of the coating are much higher in the early stages of drying for coatings based on the new Fast Cure technology. These improved properties are, of course, accompanied by very good outdoor durability.

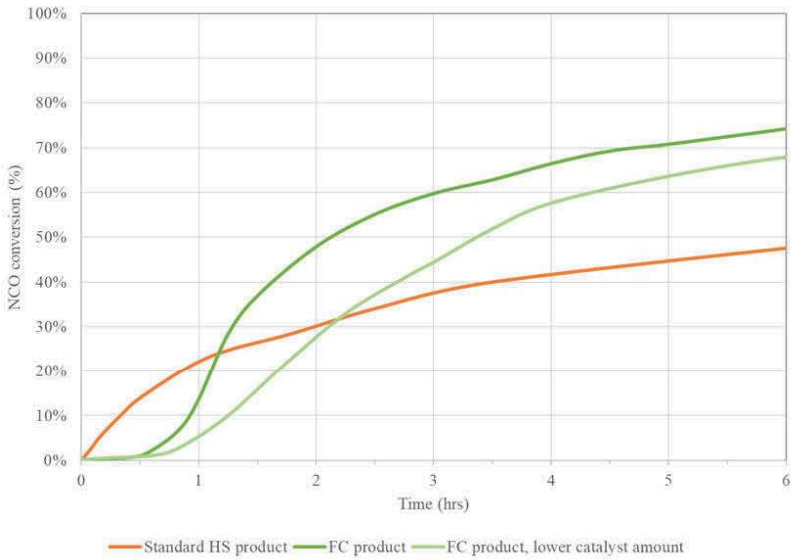

Figure 3: Isocyanate conversion versus time determined with infrared spectroscopy

Table 4: Comparison of Tg and crosslinking density between a standard system and the new Fast Cure technology

	Fast Cure technology		Standard system	
	1 day RT	7 days RT	1 day RT	7 days RT
Tg (max tan δ, °C)	46	47	38	43
Crosslinking density (mmol/cm^3)	0.91	0.91	0.66	0.78

DUROFTAL® FC High Solid polyester polyols for top coat application

Two DUROFTAL® FC grades are available, DUROFTAL® FC 9511/80BAC and DUROFTAL® FC 2828/75BAC, delivered at respectively 80% and 75% solids in butyl acetate. Their main features are described in Table 5.

Related to their polymer design, both grades, when crosslinked with aliphatic polyisocyanate hardeners, provide excellent outdoor durability.

Comparative data of white pigmented topcoats based on the new DUROFTAL® FC polyester grades versus high solid acrylic references, SETALUX ® FC 1923 and SETALUX ® 1910, are summarised in Tables 6 and 7. All formulations were tested with Desmodur ultra N 3300 hardener (NCO/OH: 1.0); DBTL catalyst level was optimised for each formulation for best overall performance; pigment (TiO_2)/binder ratio was 0.8.

In comparison to HS acrylic polyols, the FC polyesters provide further increased paint solids and lower paint VOC content; DUROFTAL® FC 9511 and SETALUX® FC 1923 demonstrate outstanding drying performance at ambient and slightly forced cure conditions with outstanding early hardness for the former. DUROFTAL® FC 2828 gave the lowest paint VOC content and the best MEK resistance after slightly forced or ambient cure. In addition, it was

Table 5: DUROFTAL® FC high solids grades

DUROFTAL® FC 9511/80BAC	4.5% OH	Topcoats with outstanding early hardness at ambient cure	- Long pot life even at high catalyst levels - Outstanding drying and early hardness at ambient conditions - Low paint VOC <300g/L (1000 mPa.s) - Excellent mechanical properties - Excellent weathering resistance
DUROFTAL® FC 2828/75BAC	6.5% OH	- Direct to metal (DTM) monocoats with superior performance on smooth untreated steel - Highly chemical and solvent resistant topcoats	- Long pot life even at high catalyst levels - Superior early hardness upon forced cure - Lowest paint VOC < 250g/L (1000 mPa.s) - High chemical and solvent resistance - Excellent UV and corrosion resistance

Table 6: Paint data and drying performances of DUROFTAL® FC and SETALUX® FC grades versus high solids acrylic references

		DUROFTAL® FC 9511/80BAC	DUROFTAL® FC 2828/75BAC	SETALUX® FC 1923 BA-75	SETALUX® 1910 BA-75
OH% on solid resin		4.5	6.5	3.6	3.6
Form of delivery		80% in BAC	75% in BAC	75% in BAC	75% in BAC
Dynamic Viscosity		3.0 - 4.0 Pa.s	2.5 - 4.5 Pa.s	3.3 - 12 Pa.s	3.9 - 11 Pa.s
Catalyst level (%)	DBTL on solid polyol	**0.32**	**0.35**	**0.21**	0.05
Paint solids (%)	at 30s (DIN-4)	74.3	**77.7**	71.4	71.8
VOC (g/L)	at 30s (DIN-4)	339	**296**	368	359
Pot life (h)	doubling of viscosity	**4.2**	**2.9**	**3.5**	1.5
Tack-free time (h)	152µm wet on glass	**3.3**	**3.5**	**2.5**	4.5
Drying recorder; 152µm wet on glass plate					
23°C	Stage 1 (h)	1.2	1.2	0.6	0.3
	Stage 2 (h)	2.1	2.3	1.8	2.0
	Stage 3 (h)	2.5	3.5	3.0	5.0
	Stage 4 (h)	**3.4**	**7.0**	**5.7**	12.0

Table 7: Hardness development and MEK resistance of DUROFTAL® FC and SETALUX® FC grades versus high solids acrylic references

		DUROFTAL® FC 9511/80BAC	DUROFTAL® FC 2828/75BAC	SETALUX® FC 1923 BA-75	SETALUX® 1910 BA-75
Pendulum hardness acc. König (s); 200µm wet on glass plate (approx. 55-60µm dft)					
23°C	5h	**21**	9	14	/
	24h	**71**	**47**	**57**	33
	168h	111	95	107	101
30min/ 50°C	1h	**44**	12	20	/
	24h	**95**	**79**	**76**	55
	168h	121	115	115	111
30min/ 80°C	1h	**119**	**113**	**93**	68
	24h	**121**	115	112	104
	168h	121	149	142	152
MEK double-rubs; 200µm wet on glass plate (approx. 55-60µm dft)					
23°C	168h	131	**> 200**	130	98
30min/ 50°C	168h	174	**> 200**	142	110
30min/ 80°C	168h	> 200	> 200	137	> 200

found that DUROFTAL® FC 2828 displayed superior corrosion resistance on various relevant substrates, like Gardobond OC, Gardobond WH 6800 OC, sandblasted steel and even also on more challenging substrates like untreated smooth steel. The corrosion and humidity resistance performance are shown in Figure 4 and Table 8. A red pigmented topcoat was prepared based on DUROFTAL® FC 2828, cured for 30 min at 80°C and left for 1 week at room temperature. Subsequently, panels were exposed to salt spray testing for 1000 hours. Hardly any blisters were observed, accompanied by very low delamination (cut) and still perfect adhesion after 1000 hours salt spray.

Formulation advice

The chemistry of FC resins allows usage of higher levels of tin catalysts to speed up curing, while still keeping a good pot life. Too high levels of tin catalyst might, however, lead to (too) fast curing of the coating surface. This can result in more solvent retention with the risk of dieback and, in the end, lower coating hardness. Therefore, it is important to choose the right balance between the amount of catalyst and/or slow solvents and take into account the preferred application (e.g. RT vs 60°C cure). Safety and health concerns about the use of (high concentrations of) tin catalysts, moreover, must also not be forgotten: in this respect, application of FC cure technology to tin-free systems is currently under study.

In combination with the recommended higher levels of tin catalyst, bases and more specifically amines can also be used to fine-tune performances: higher concentrations of such components will result in reduced tack-free time, but also in shorter pot life and lower end hardness. Addition of acidic components, on the other hand, will result in prolonged pot life, longer drying times and lower early hardness.

When choosing formulation components, paint formulators should consider that their acidic or basic properties can impact the coating performances. This is illustrated by the pot life results reported in Figure 5, concerning the use of different dispersants in a top coat formulation based on DUROFTAL® FC 9511/80BAC.

Figure 4: Salt spray testing on red pigmented topcoat based on DUROFTAL® FC 2828 for 1000 hours

Table 8: Salt spray and humidity resistance data of red pigmented topcoat based on DUROFTAL® FC 2828

	Salt spray resistance[1]		Humidity resistance[2]
	Gardobond OC		
	97 µm		95 µm
	Delamination[3] (cut, mm)	Blisters (area)	Blisters (area)
240h	6	0	0
480h	9	0	4(S2)
720h	5	0	4(S2)
1000h	6	3(S2)	4(S2)
Adhesion after 1000h			Gt 0
	Gardobond WH 6800 OC		
	116 µm		110 µm
	Delamination[3] (cut, mm)	Blisters (area)	Blisters (area)
240h	3	0	0
480h	3	0	0
720h	3	0	0
1000h	4	0	0
Adhesion after 1000h			Gt 0
	Sandblasted steel		
	121 µm		126 µm
	Delamination[3] (cut, mm)	Blisters (area)	Blisters (area)
240h	3	0	0
480h	4	0	0
720h	5	0	0
1000h	3	0	0
Adhesion after 1000h			Gt 0

[1] acc. DIN EN ISO 9227
[2] acc. DIN EN ISO 6270-2
[3] scratched immediately after SS chamber, no recovery

Conclusions

In general, all resins in the new allnex Fast Cure product range have the following features:

- fast to ultrafast drying at room temperature or elevated temperature for shorter cycle times and increased profitability,
- exceptionally long pot life and working time,
- excellent early hardness,
- excellent appearance, and

all this combined with very good chemical resistance, mechanical properties and outdoor durability.

The combination of these special features and advantages of the Fast Cure product range is also visualised in the spider diagram of Figure 6. Here, the general advantages of all the Fast Cure products become evident, but this diagram also shows that each of the Fast Cure resins comes with its own further special benefits, allowing paint formulators to optimise their paints according to the needs of their specific application by the blending of Fast Cure resins.

Initially developed for clear coat applications in vehicle refinish, the new Fast Cure technology has also demonstrated exceptional performances in pigmented top coat applications for general industrial and DTM applications. Paint formulators in the flooring business can also benefit from the advantages Fast Cure is offering: very good results have been obtained when flooring paints were formulated with SETATHANE® FC 1135. In addition, Fast Cure products have been demonstrated to give all the above advantageous features in clear coat formulations for OEM plastics and wood. Especially in OEM plastic application, Fast Cure products can also be used to contribute to more environmentally friendly processes, enabling lower temperature curing conditions and saving energy consumed by the ovens. Application of Fast Cure products can be further extended to other markets, such as ACE and marine & protective. FC-compatible sag control agent-modified resins for OEM applications are also currently under development.

For questions on how your business can profit from the benefits offered by the allnex Fast Cure product range, please contact a member of the allnex sales team or visit the allnex website.

Table 9: Top coat formulations based on DUROFTAL® FC 9511/80BAC

DUROFTAL® FC 9511/80BAC		125	125	125
BAC		25	25	25
Acidic dispersant	ca. 3% active substance on pigments		6.04	
Basic dispersant	ca. 3% active substance on pigments			12.08
Kronos 2310	pH: 7 (10% in water)	120.8	120.8	120.8
Cravallac extra	ca. 1.0% on paint	3.6	3.67	3.75
ADDITOL® VXL 4951 N	ca. 0.3% on paint	1.1	1.1	1.1
MODAFLOW® RESIN	ca. 0.5% on paint	1.8	1.84	1.88
Tinuvin 123	ca. 2% on TRS	3.02	3.02	3.02
Tinuvin 1130	ca. 1% on TRS	1.51	1.51	1.51
Metatin Catalyst 712 ES/1X		25	25	25
Desmodur N 3300	NCO/OH = 1	51	51	51
BAC	adjust to 30s (DIN-4)	33.57	24	47.81

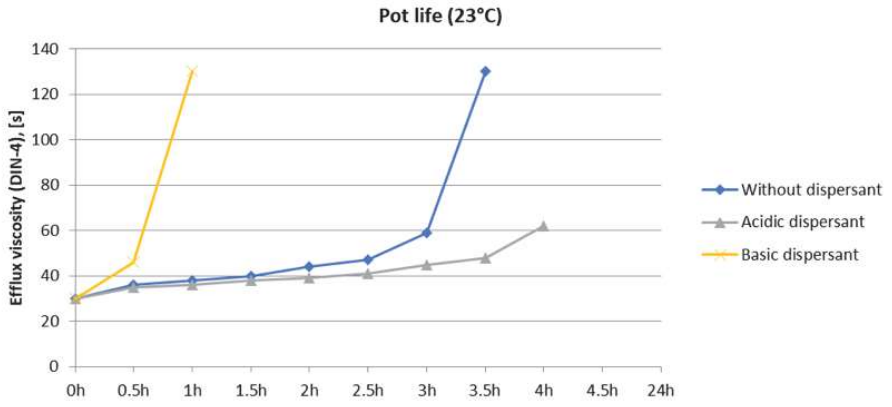

Figure 5: Impact of dispersant on pot life of top coat formulations based on DUROFTAL® FC 9511/80BAC

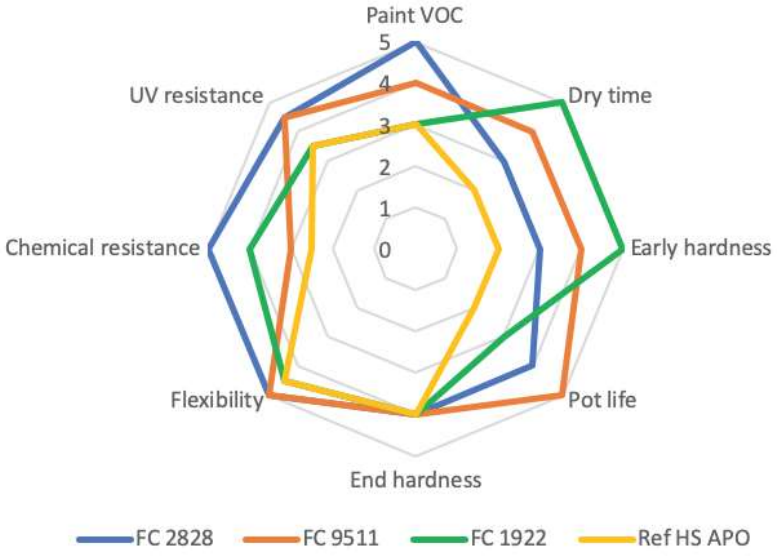

Figure 6: Spider diagram summarising the advantages and special features of high solids Fast Cure resins