



Encapsulated EPFR-APP series applied to intumescent fire proof coating

Eco-Friendly Halogen-free Flame Retardant

Product Form: white powder



Version 1.4
2014-10

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Testing methods and standards of APP on the market that we know

| Items | Standard/method | Test equipment |
|-----------------------|-----------------------|-------------------------------|
| pH value | 25°C , 10% suspension | pH Meter |
| Water content | 105°C/2h | Drying Oven |
| Water solubility | 25°C , 10% suspension | High-speed mixer, Drying Oven |
| Particle size | ASTM E112-1996 | Malvern Mastersize 2000 |
| Viscosity | ASTM D445-06 | Viscosity Checker |
| TGA | ASTM D3850-12 | TA Q500 |
| Polymerization degree | ³¹ P NMR | Bruker-400 NMR |

Comparison on TDS data of MF encapsulated APP

| Item code | Phosphorus content (%) | Nitrogen content (%) | pH | Viscosity (mPa·s) | Polymerization degree | Water content (%) | Solubility (%) | Particle size D ₅₀ (μm) |
|-------------|---------------------------|-------------------------|---------|----------------------|--------------------------|----------------------|----------------|--|
| EPFR-APP262 | ~29 | ~16.5 | 5.5~7.5 | ≤20 | ≥1000 | ≤0.5 | ≤0.04 | ~10 |
| EPFR-APP263 | ~29 | ~16.5 | 5.5~7.5 | ≤20 | ≥1000 | ≤0.5 | ≤0.04 | ~20 |
| APP-C2 | 29~31 | 15~17 | 6.5~8.5 | ≤20 | ≥1000 | ≤0.5 | ≤0.04 | ~20 |
| APP-J2 | ~30 | -- | 4.0~7.0 | ≤5 | ≥1000 | ≤0.5 | ≤0.04 | ~18 |
| APP-B2 | ~29 | -- | 6.0~7.0 | -- | ≥1000 | ≤0.5 | ~0.01 | ~18 |

Comparison on basic data of MF encapsulated APP

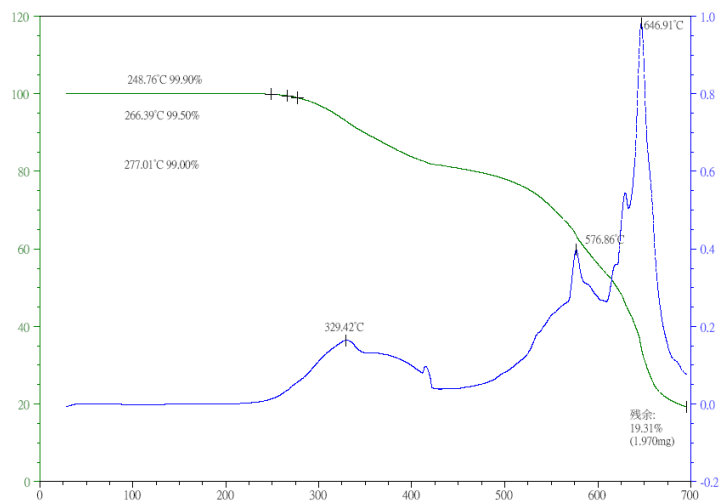
| Item code | Whiteness (%) | pH value | Viscosity (mPa·s) | Water content (%) | Water solubility (%) |
|--------------------|---------------|----------|-------------------|-------------------|----------------------|
| EPFR-APP262 | 94.7 | 6.38 | 6.5 | 0.14 | 0 |
| EPFR-APP263 | 92.4 | 6.90 | 6.5 | 0.04 | 0.02 |
| APP-C2 | 86.7 | 7.17 | 9.2 | 0.52 | 0.01 |
| APP-J2 | 87.7 | 6.56 | 9.2 | 0.36 | 0.03 |
| APP-B2 | 93.2 | 5.43 | 10.2 | 1.03 | 0.03 |

Comparison on basic data of MF encapsulated APP

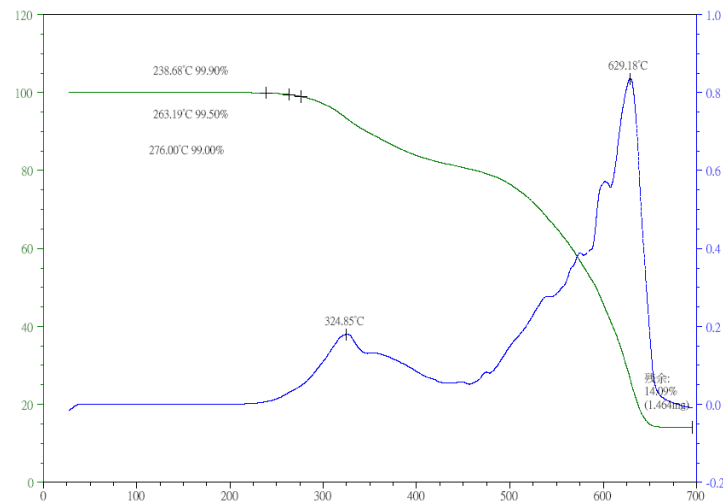
| Item code | Particle size(μm) | | Polymerization Degree | Small molecule(%) | TGA ($^{\circ}\text{C}$) | | | | |
|--------------------|--------------------------------|-----------------|-----------------------|-------------------|----------------------------|--------|--------|--------|--------|
| | D ₅₀ | D ₉₀ | | | 0.1% | 0.5% | 1.0% | DTG1 | DTG2 |
| EPFR-APP262 | 12.21 | 23.48 | 3333 | 0.21 | 248.76 | 266.39 | 277.01 | 329.42 | 576.86 |
| EPFR-APP263 | 20.62 | 36.94 | 10000 | 0.21 | 238.68 | 263.19 | 276.00 | 324.85 | 629.18 |
| APP-C2 | 18.33 | 34.92 | 3333 | 0.11 | 73.59 | 237.60 | 254.65 | 304.23 | 534.66 |
| APP-J2 | 20.71 | 38.87 | 1818 | 0.36 | 49.64 | 236.66 | 260.11 | 304.92 | 539.77 |
| APP-B2 | 19.26 | 38.74 | 4000 | 0.30 | 30.18 | 240.44 | 247.91 | 348.99 | 534.45 |

Comparison on TGA curves of MF encapsulated APP

Preniphor™ EPFR-APP262

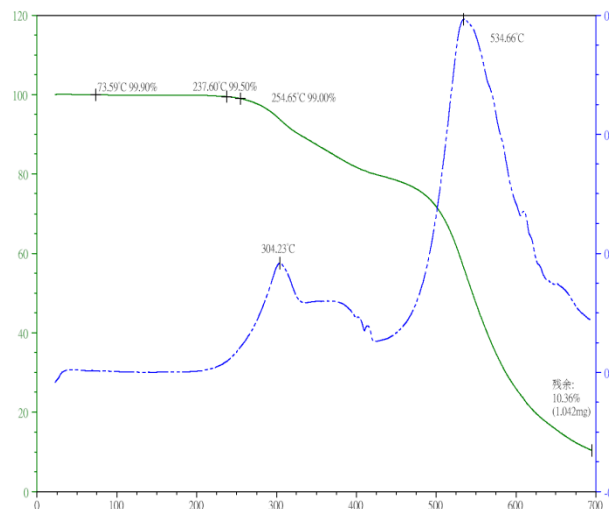


Preniphor™ EPFR-APP263

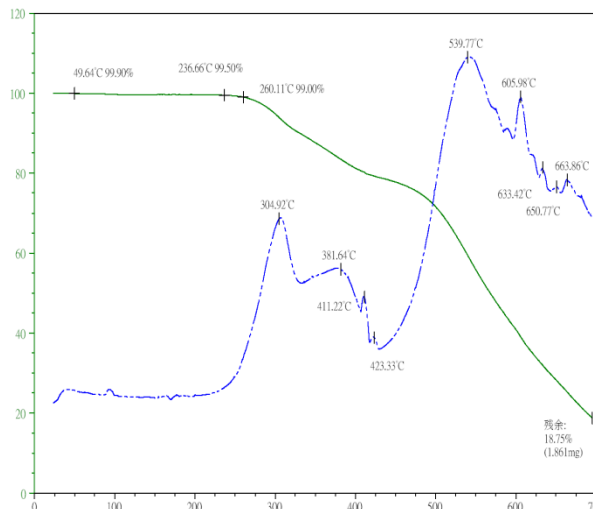


Comparison on TGA curves of MF encapsulated APP

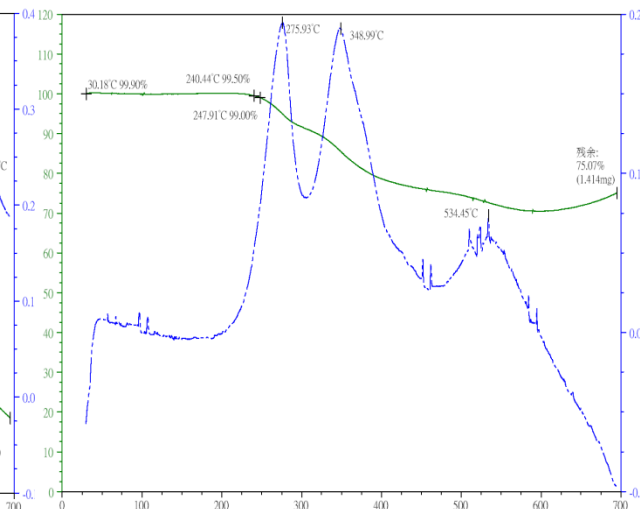
APP-C2



APP-J2



APP-B2

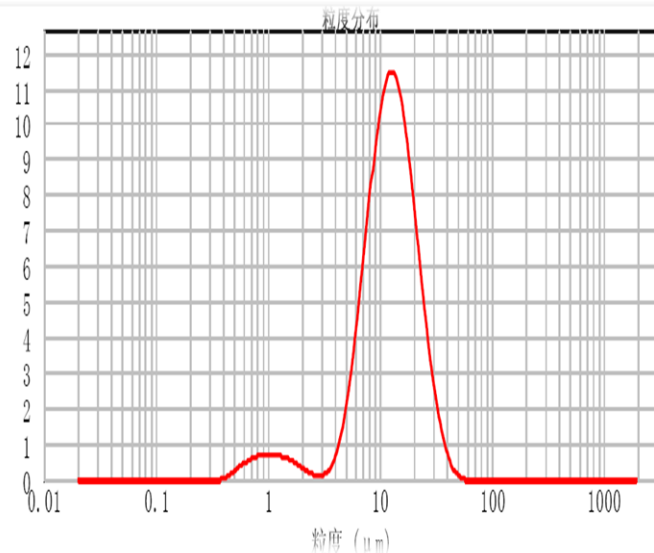


Comparison on particle size of MF encapsulated APP

Preniphor™ EPFR-APP262

Particle size distribution

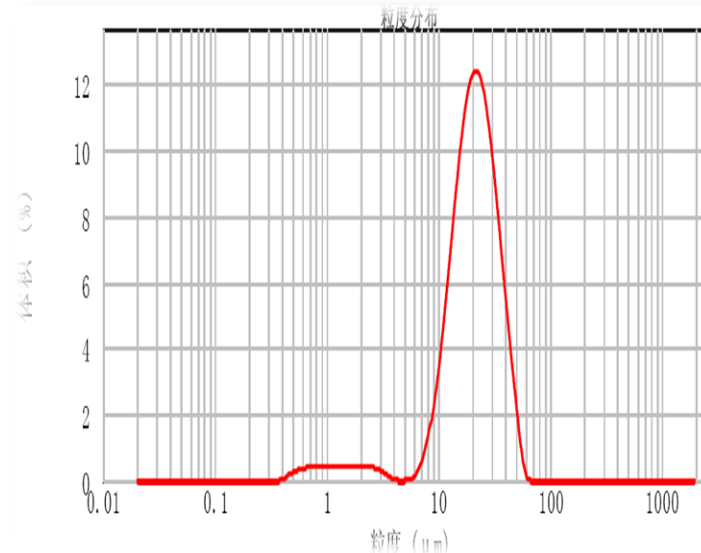
| | | | |
|-----------------|----------|-----------------|----------|
| D ₁₀ | 5.65 μm | D ₅₀ | 12.21 μm |
| D ₉₀ | 23.48 μm | D ₉₈ | 33.32 μm |



Preniphor™ EPFR-APP263

Particle size distribution

| | | | |
|-----------------|----------|-----------------|----------|
| D ₁₀ | 10.26 μm | D ₅₀ | 20.62 μm |
| D ₉₀ | 36.94 μm | D ₉₈ | 47.85 μm |

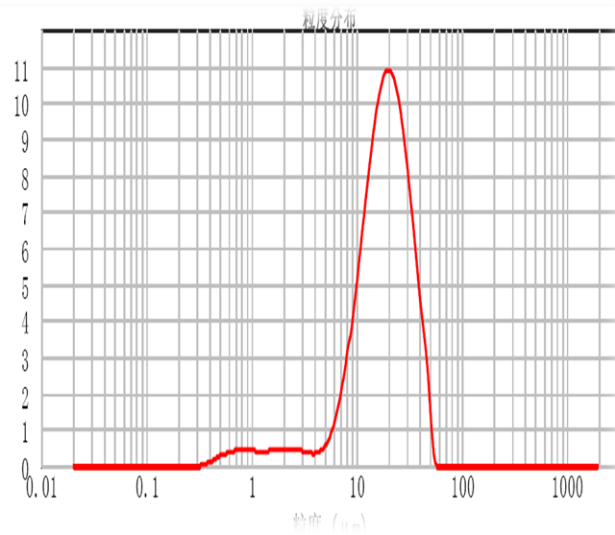


Comparison on particle size of MF encapsulated APP

APP-C2

Particle size distribution

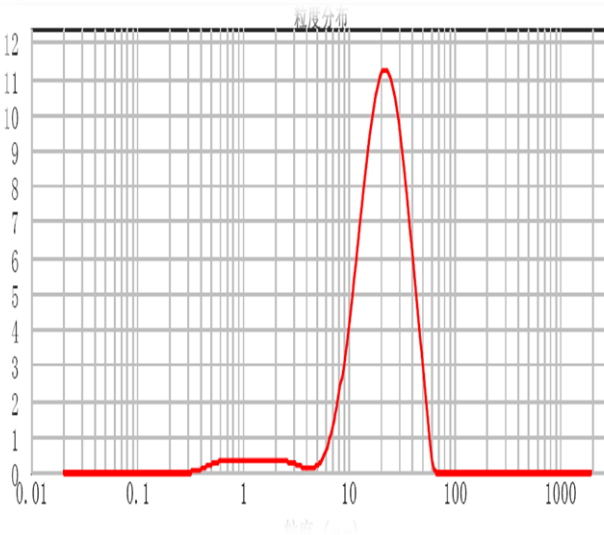
| | | | |
|-----------------|----------|-----------------|----------|
| D ₁₀ | 7.64 μm | D ₅₀ | 18.33 μm |
| D ₉₀ | 34.92 μm | D ₉₈ | 45.35 μm |



APP-J2

Particle size distribution

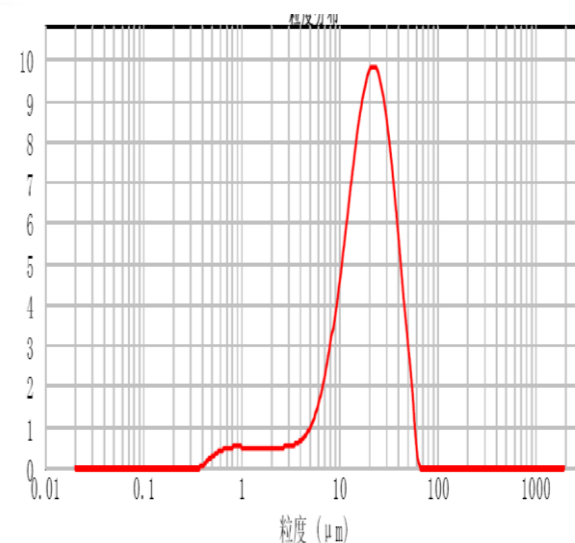
| | | | |
|-----------------|----------|-----------------|----------|
| D ₁₀ | 9.61 μm | D ₅₀ | 20.71 μm |
| D ₉₀ | 38.87 μm | D ₉₈ | 50.47 μm |



APP-B2

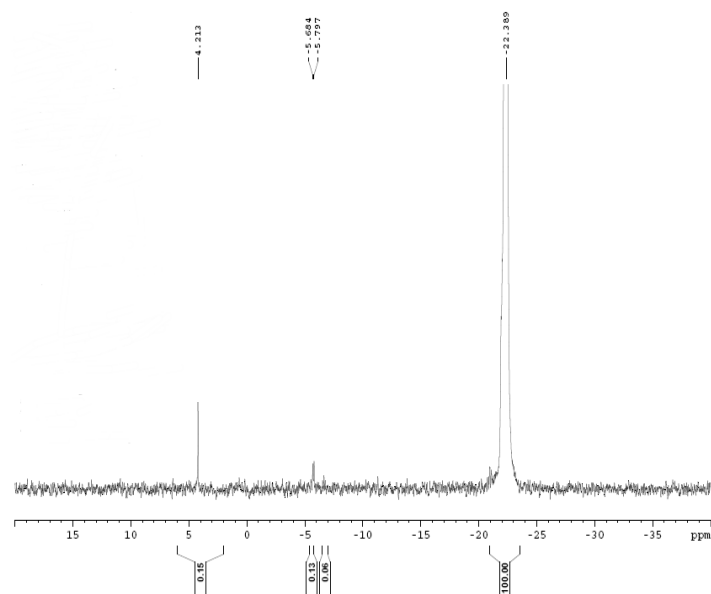
Particle size distribution

| | | | |
|-----------------|----------|-----------------|----------|
| D ₁₀ | 6.51 μm | D ₅₀ | 19.26 μm |
| D ₉₀ | 38.74 μm | D ₉₈ | 51.31 μm |

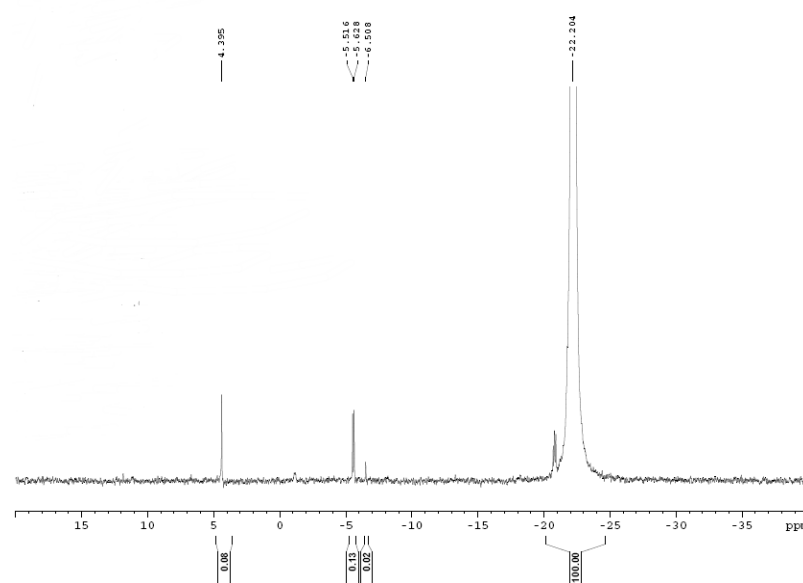


Comparison on P³¹ NMR of MF encapsulated APP

PreniphorTM EPFR-APP262



PreniphorTM EPFR-APP263

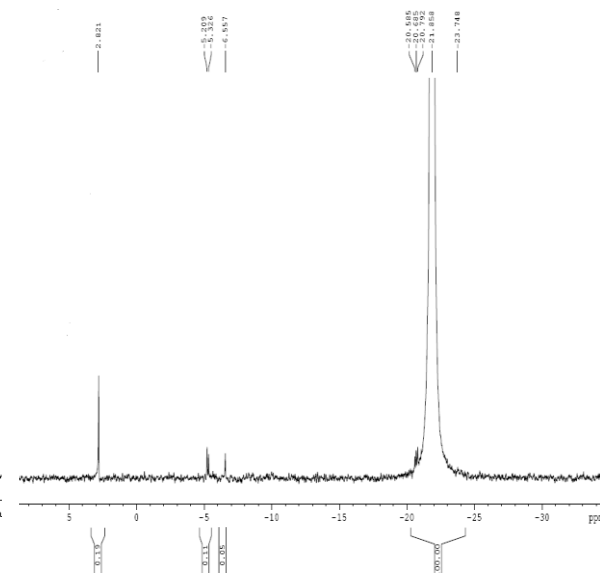
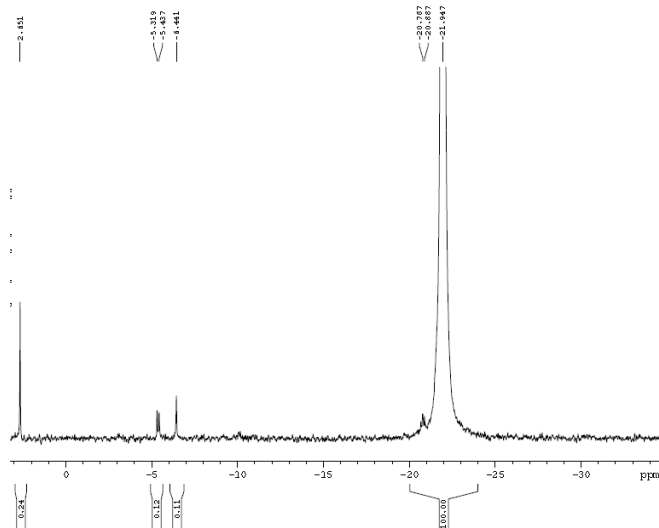
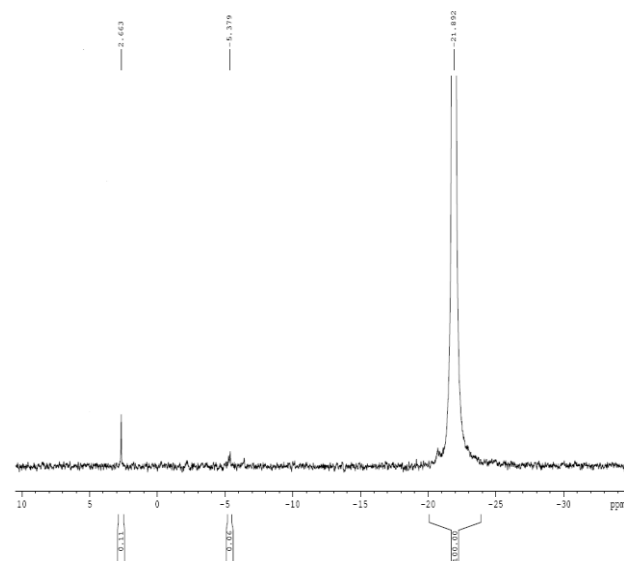


Comparison on P^{31} NMR of MF encapsulated APP

APP-C2

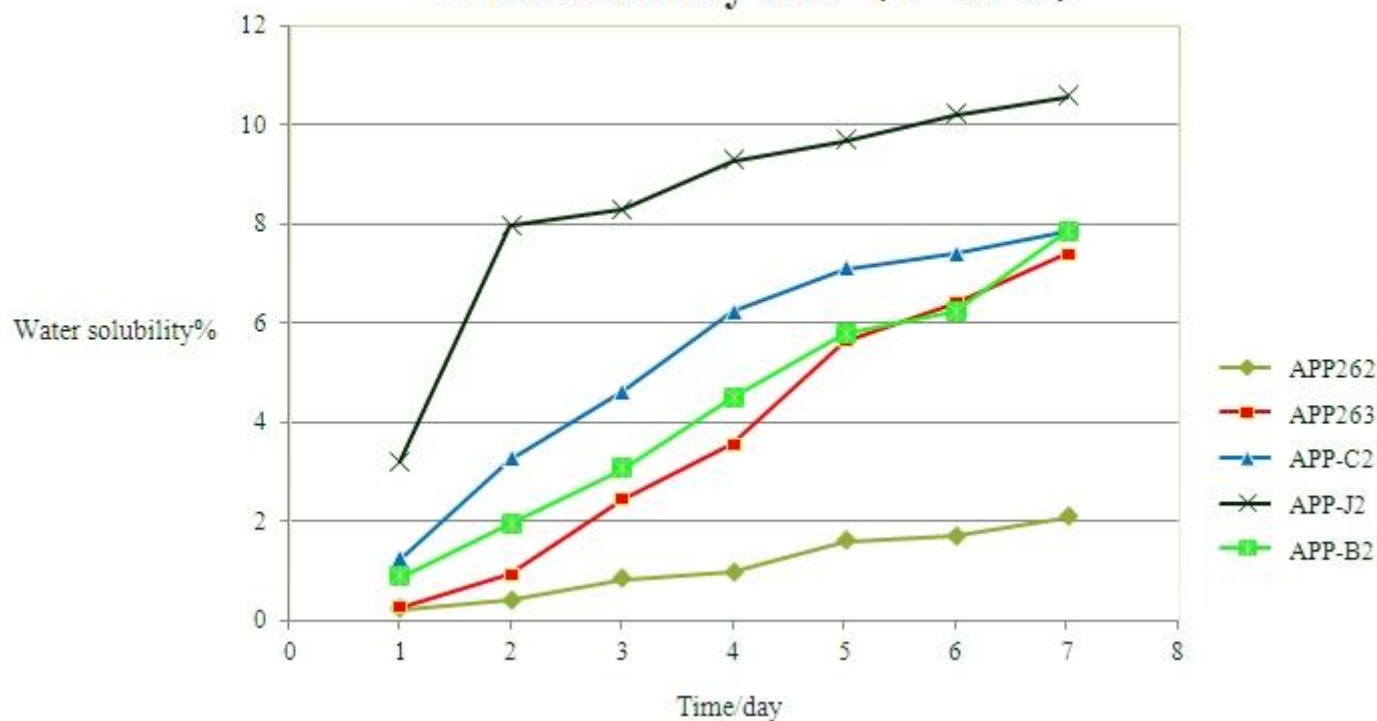
APP-J2

APP-B2



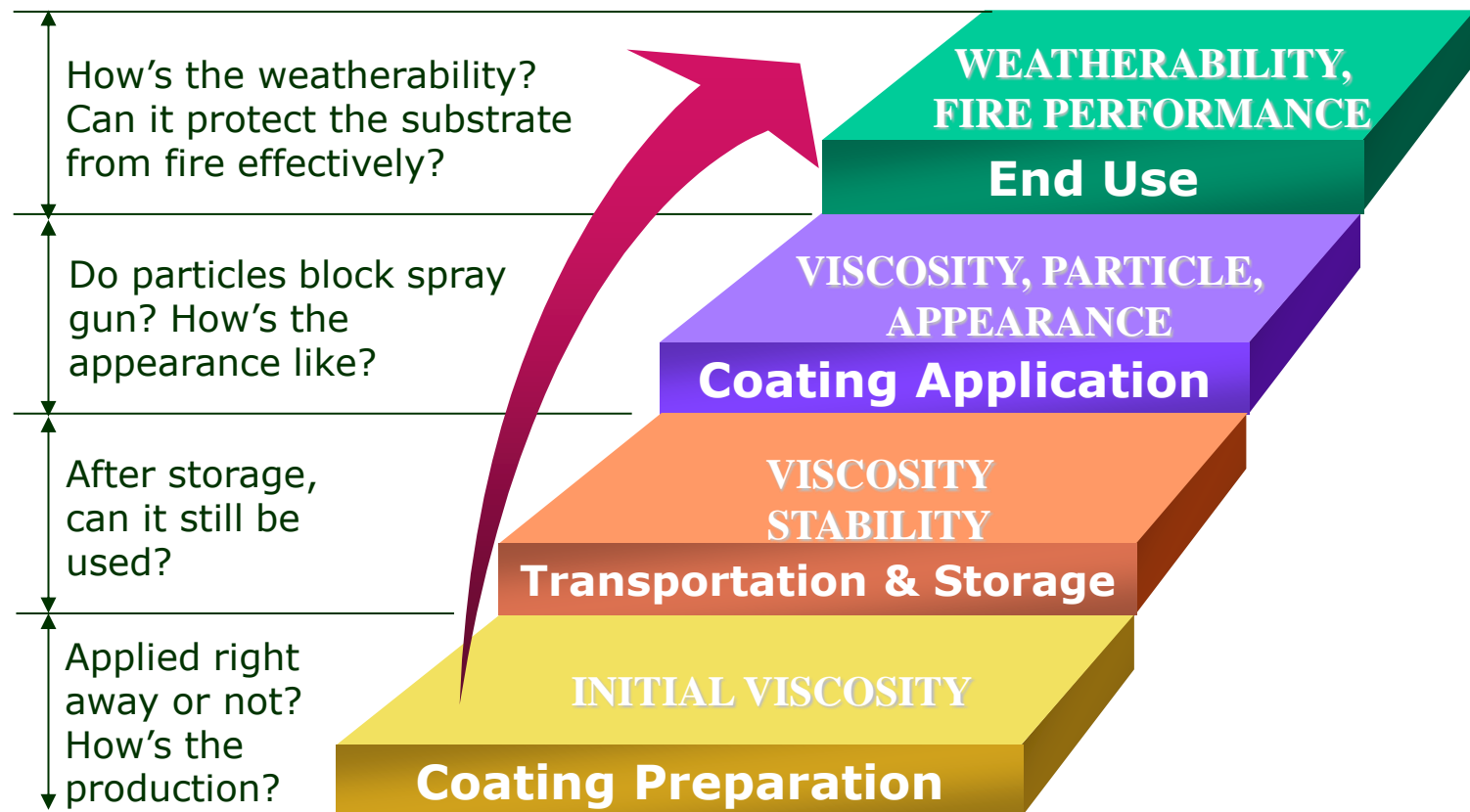
Comparison on water resistance of MF encapsulated APP

Water solubility test (T=80℃)

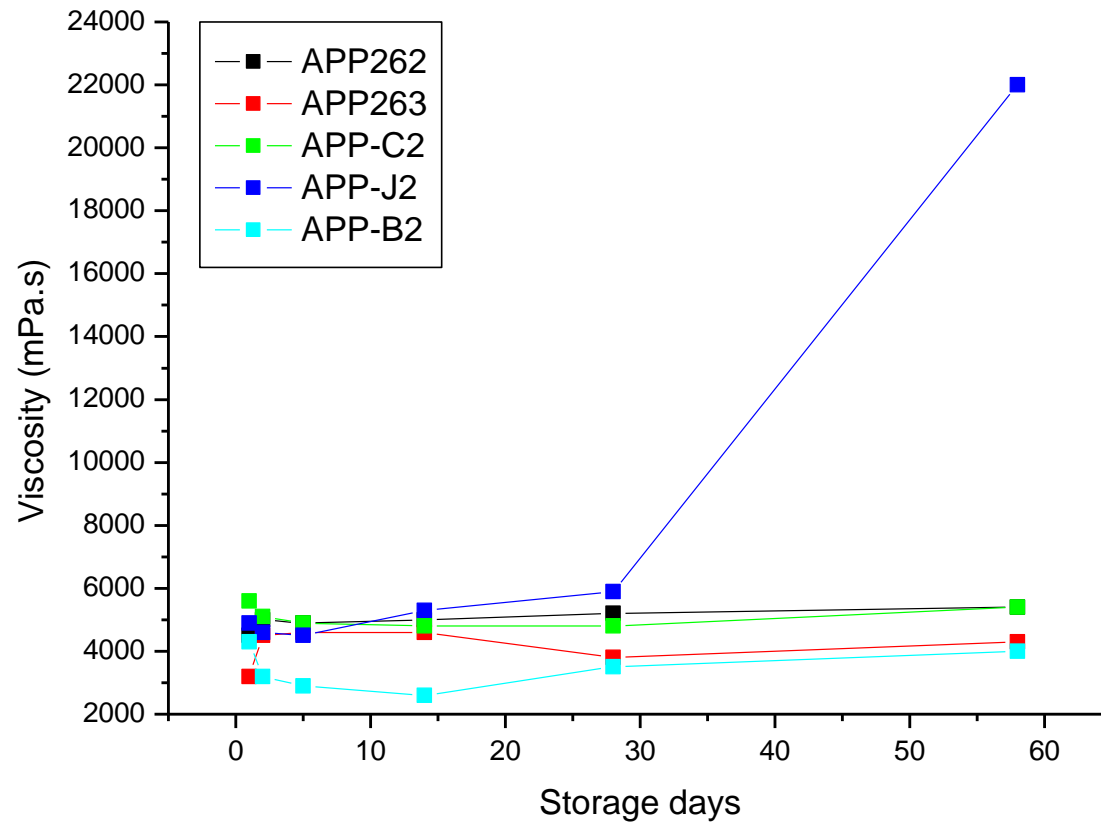


Test method: Make 10% saturated solution with 100g powder and 900ml water, then put it into 80℃ thermostat water bath, take 50ml sample every 24h by dispersing solution evenly. We can get the water solubility after centrifugation, suction filtration, drying and weighing.

Coating's life and APP



Viscosity storage stability of MF encapsulated APP in latex



Stimulate
storage

Sample preparation: Mix 50% APP with acrylic latex (water-based, solid content 40%), and then test its viscosity with time change to evaluate APP's viscosity storage stability.

Test equipment and Sample preparation

✓ Fire performance testing equipment



Fire-proof Performance of Coating Quick-Test Equipment



Real-time temperature curve of furnace and plate backing (Green: plate backing, Red: furnace)

Remarks: The quick-test equipment is from Beijing Institute of Aeronautical Materials.

Fire performances are characterized by char morphology, temperature curve and fire resistant time.

Sample preparation

✓ Samples

| Samples | Note |
|--------------|--|
| Blank Sample | Coating without adding APP, for reference |
| EPFR-APP 262 | Coating that adds EPFR-APP 262 from PRESAFER |
| EPFR-APP 263 | Coating that adds EPFR-APP 263 from PRESAFER |
| APP-C2 | Coating that adds APP from German company C |
| APP-J2 | Coating that adds APP from China company J |
| APP-B2 | Coating that adds APP from German company B |

✓ Sample preparation

- ◆ Paint the undercoat (thickness is about 0.1mm) on the surface of a clean steel plate.
- ◆ Paint fire-proof coating after the undercoat dried. Calculate the specific content of fire-proof coating on the steel plate according to the solid content. Making sure that the thickness of the dry coating on the steel plate is $1.00\text{mm} \pm 0.05\text{mm}$. The thickness of the fire-proof coating should be as even as possible .
- ◆ The specimen with FR coating should be air-dry after 7 days, or at least after 1-2 days, and then get dried below $40\text{ }^{\circ}\text{C}$ for 1-2days, until the actual thickness is within 0.05mm of the calculation value.
- ◆ Measure the thickness of the coating layer and record.

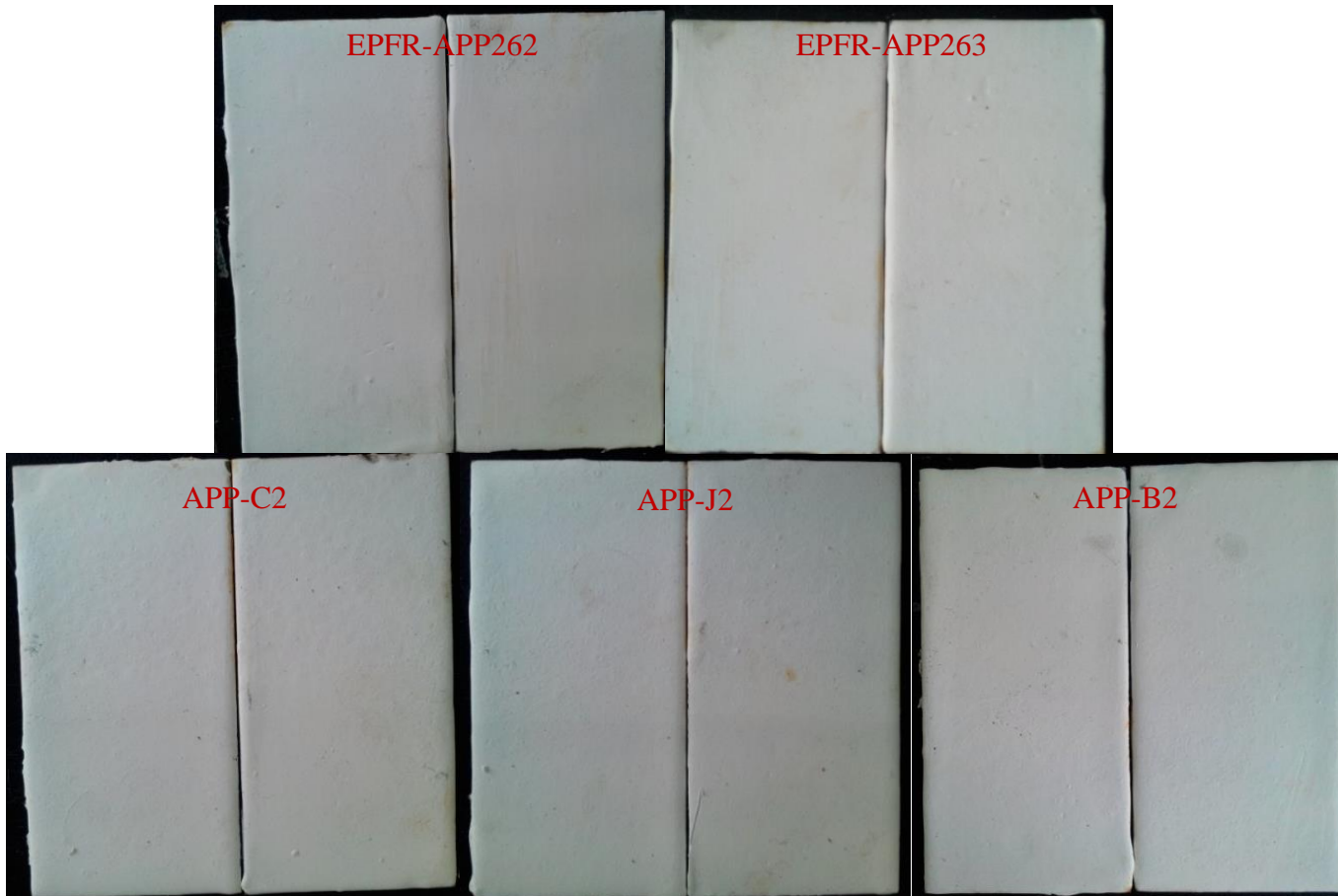
Solventborne acrylic intumescent flame retardant coating

✓ Typical formula

| Raw materials | Loading (wt %) |
|-----------------------------------|-------------------|
| Acrylic resin (Pliolite AC80/AC4) | 10-12 |
| Ammonium polyphosphate | 27-29 |
| Pentaerythritol (Charmor® PM40) | 10-12 |
| Melamine (Industrial grade) | 9-12 |
| Chlorinated paraffin (CP-70) | 3-5 |
| Titanium dioxide (R103) | 8-12 |
| Other filler and additives | 3-5 |
| Solvent | 20-25 |
| Total content | 100 |

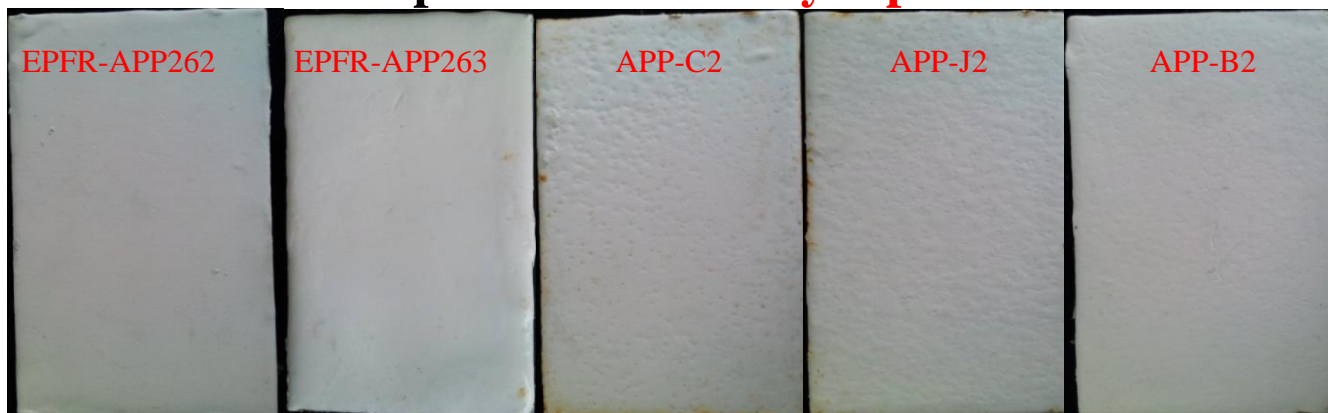
Solventborne acrylic intumescent flame retardant coating

Photos of coated plates- Immersed in still water for 24h

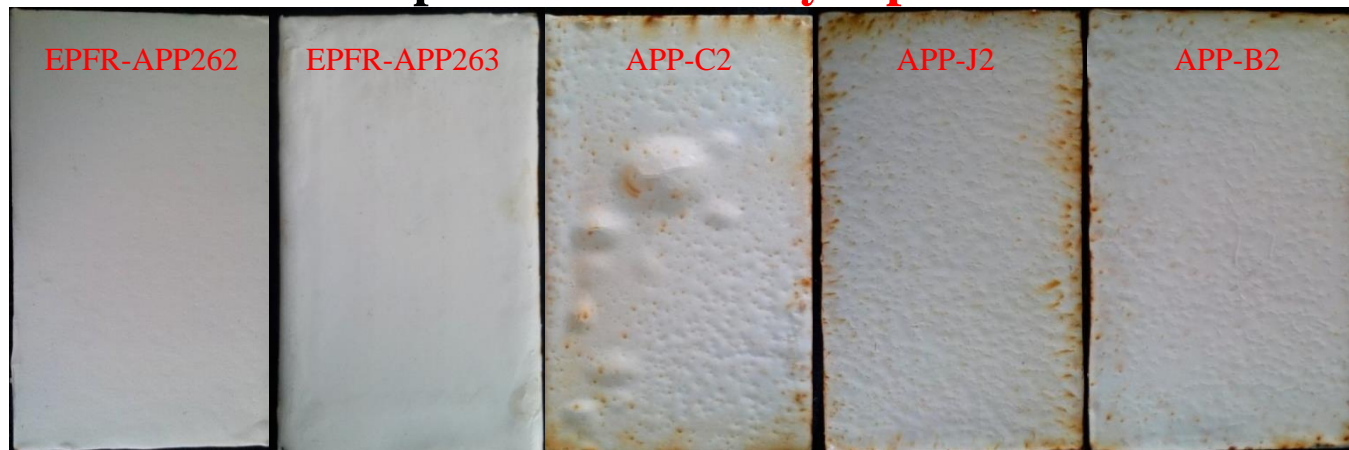


Solventborne acrylic intumescent flame retardant coating

Photos of coated plates - Washed by tap-water for 24h

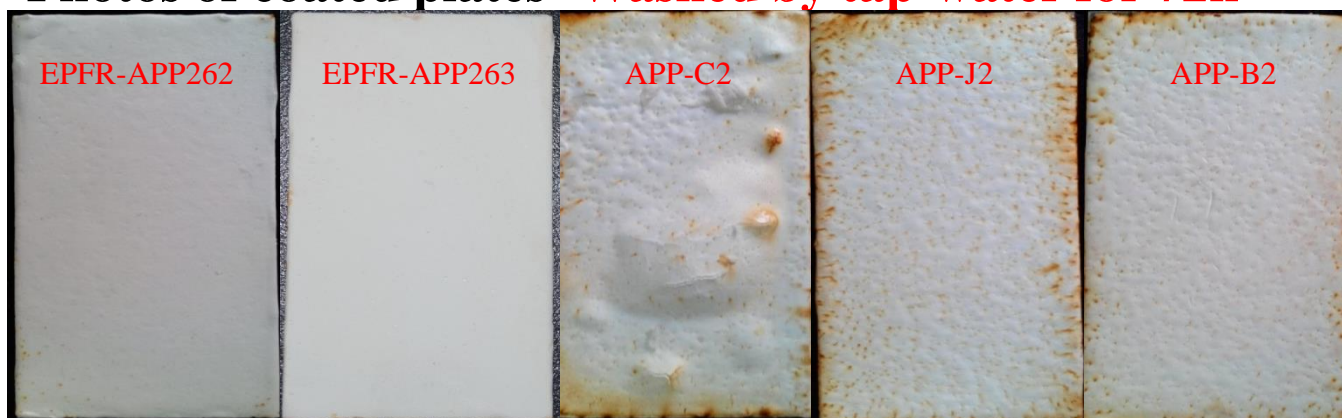


Photos of coated plates - Washed by tap-water for 48h

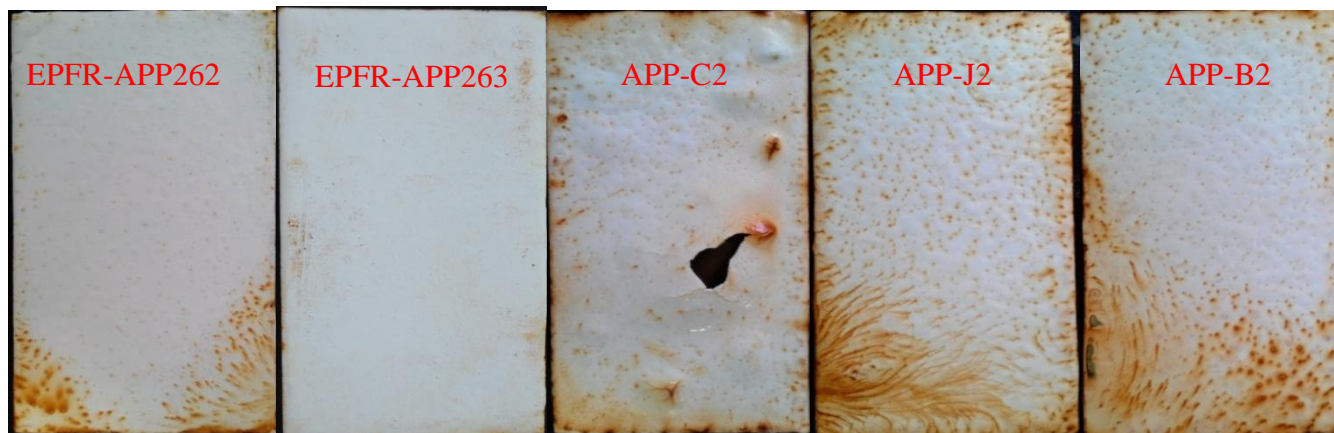


Solventborne acrylic intumescent flame retardant coating

Photos of coated plates - Washed by tap-water for 72h

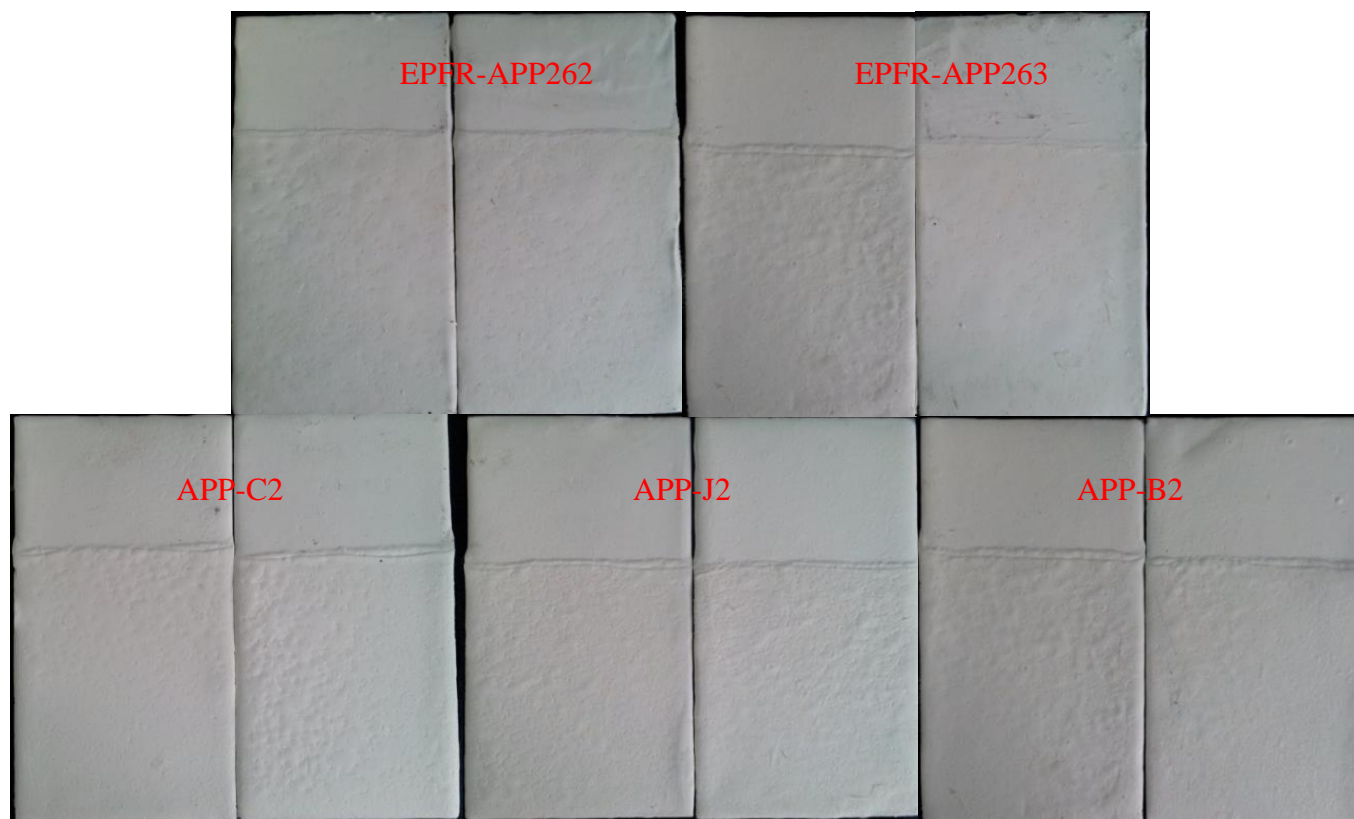


Photos of coated plates - Washed by tap-water for 120h

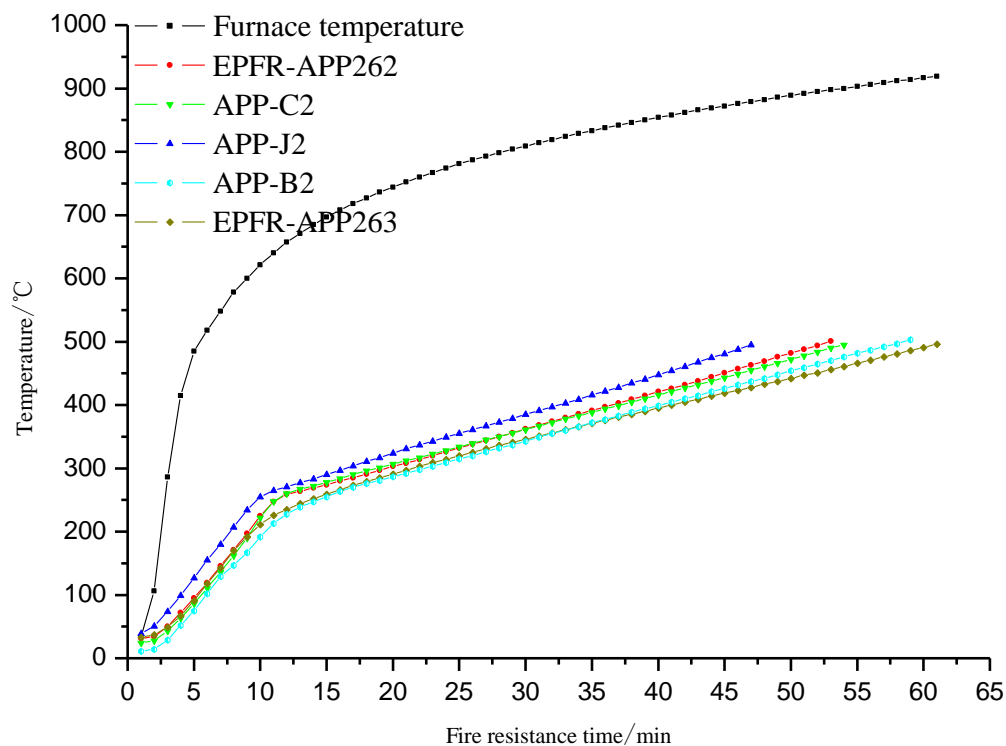


Solventborne acrylic intumescent flame retardant coating

Photos of coated plates - Immersed in 3% NaOH solution for 24h



Solventborne acrylic intumescent flame retardant coating



| Sample | Fire-resistance time |
|-------------|----------------------|
| EPFR-APP262 | 57min |
| EPFR-APP263 | 64min |
| APP-C2 | 58min |
| APP-J2 | 48min |
| APP-B2 | 59min |

Solventborne acrylic intumescent flame retardant coating

Char photos



EPFR-APP262



EPFR-APP263



APP-C2

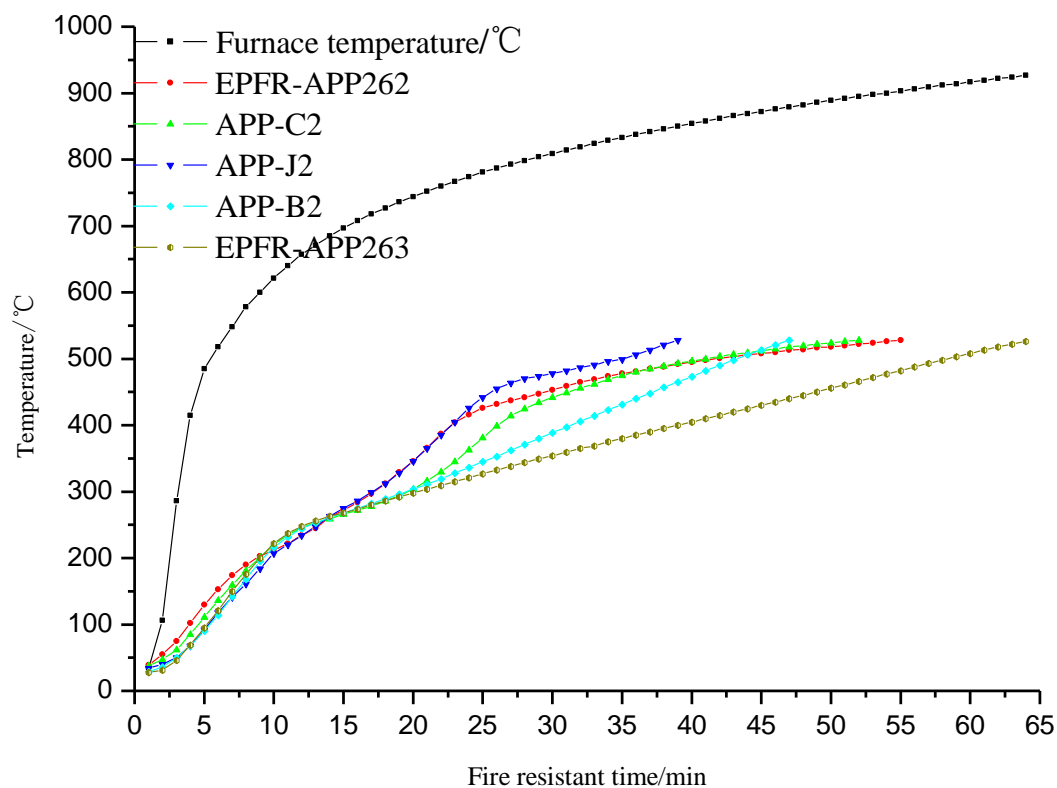


APP-J2



APP-B2

Fire performance after 1 year storage of coated plate with different APP



| Sample | Fire-resistance time |
|-------------|----------------------|
| EPFR-APP262 | 55min |
| EPFR-APP263 | 63min |
| APP-C2 | 52min |
| APP-J2 | 39min |
| APP-B2 | 47min |

Fire performance after 1 year storage of coated plate with different APP

Char photos



EPFR-APP262



EPFR-APP263



APP-C2



APP-J2



APP-B2

Solventborne acrylic intumescent flame retardant coating

- ◆ Crossbeam test (thickness of coating is 2.0mm)



| | |
|--------------------|--|
| Item code | EPFR-APP262 |
| Description | 1.7m beam |
| Char | Even and dense char, high expansion ration and no crack |

| | | |
|-------------------------|--------------------------------|------------------------------|
| Item code | Fire proof time | Final temperature |
| EPFR- APP262 | 60min | 518℃ |

Conclusion

- **Compared with other APP on the market, Preniphor™ EPFR-APP(II) has the advantages as below:**
- ❖ **High polymerization degree**
 - ❖ **Good weather-resistance**
 - ❖ **Excellent flowability and dispersity**
 - ❖ **Small content of low polymerization degree substance**
 - ❖ **Outstanding thermal stability**



Halogen free flame retardant specialist

PRESAfer

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