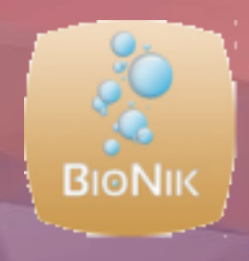


Novel end-capping method with silyl-reagent including ethylene chain



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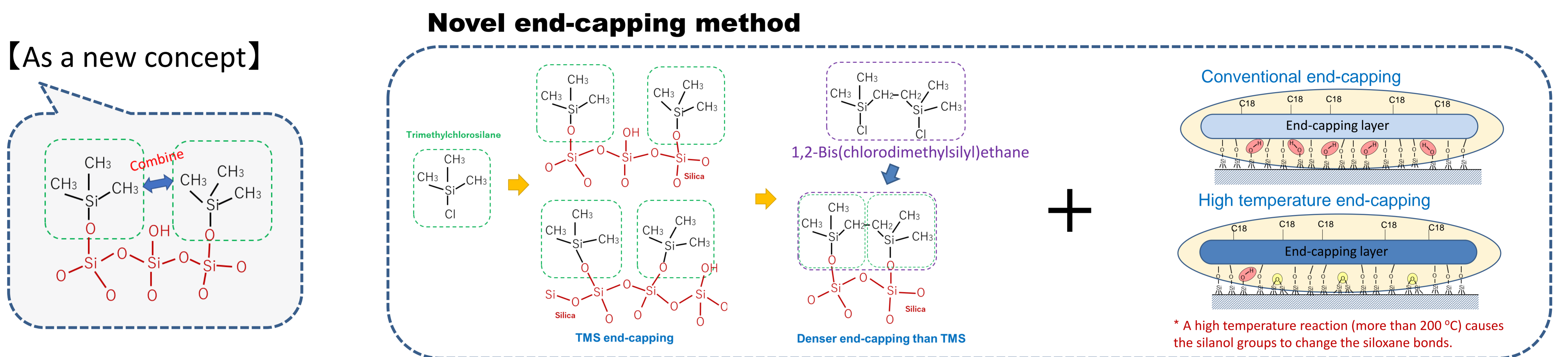
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Trimethylsilyl reagents (TMS) have been widely used as end-capping reagents for silica-based reversed-phase LC packing. Recently, difunctional silyl reagents such as hexamethyldichlorotrisiloxane have been used as end-capping reagents to reduce residual silanol groups on silica gel surface. Some brands of C18 end-capped with hexamethyldichlorotrisiloxane show an excellent peak shape for strongly basic compounds.

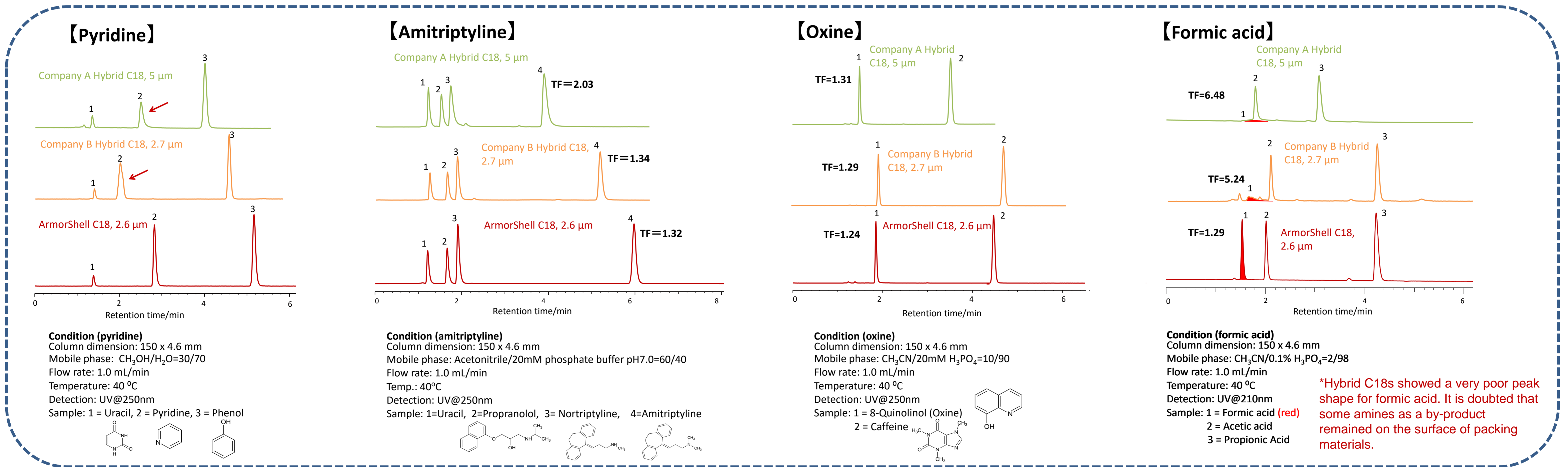
As a new concept, 1,2-bis(dimethylchlorosilyl)ethane, which was bonded the methyl groups of two trimethylchlorosilane reagents, was thought to function as a denser end-capping reagent than trimethylchlorosilane. In this study, we evaluated a reversed-phase packing with both 1,2-bis(dimethylchlorosilyl)ethane as an end-capping reagent and a high temperature reaction to convert the silanol groups into siloxane bonds. Core-shell silica was used as the silica base material for the evaluation of the two bonding techniques described above. The proposed core shell C18 phase was compared with two other core shell C18 phases for stability under both acidic and basic pH conditions and inertness towards acidic, basic and metal chelating compound. An ethylene chain is expected to provide high stability of its packing material under basic pH condition and 1,2-bis(dimethylchlorosilyl)ethane as an end-capping reagent contains an ethylene chain in the molecule, so that the proposed core shell C18 column was compared with hybrid type core shell C18 columns which were known to be more stable under basic pH conditions than a conventional C18 column.

As a result, the proposed C18 column not only exhibited almost same stability as hybrid type core shell C18 columns under basic pH conditions, but also showed much higher stability than hybrid type core shell C18 columns under acidic pH conditions. Regarding inertness towards basic and metal chelating compounds, the proposed core shell C18 column exhibited almost the same peak shape as hybrid type core shell C18 columns. However, for formic acid as an acidic compound, the proposed core shell C18 column showed a symmetrical peak while hybrid type core shell C18 columns showed a terrible tailing peak.

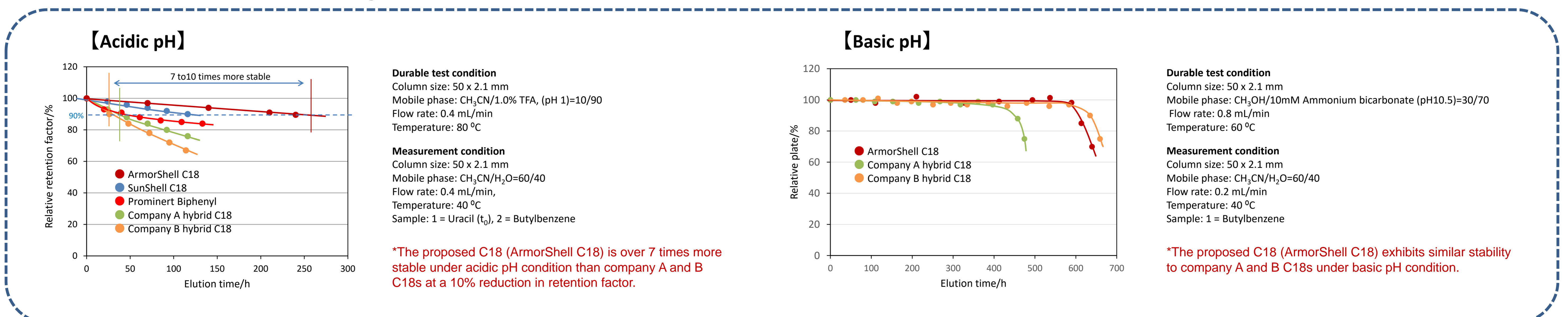


ArmorShell C18: 2.6 μm of core shell silica bonded with trifunctional C18 and end-capped using the above novel end-capping method. C18 of Company A and Company B uses hybrid core-shell silica in which ethylene chains are bonded on the silica surface. (Kinetex EVO C18 and PoroShell HPH C18)

Comparison of a peak shape



Comparison of stability under acidic and basic pH conditions



Conclusion

- ✓ The proposed end-capping method improved not only a peak shape but also stability under both acidic and basic pH conditions.
- ✓ The ethylene chains in or on the silica gel are known to improve stability under basic pH condition. But the proposed end-capping reagent including an ethylene group improved stability under not only basic pH but also acidic pH condition. It is considered that this result was not due to the effect of the ethylene chain, which has been said so far, but due to the increase in the efficiency or density of end-capping.
- ✓ Company A states that a silyl reagent with dimethylamine as a functional group was used in the patent examples. So, it is considered that the dimethylamine remaining on the silica surface adsorbed formic acid, causing severe tailing of the formic acid peak.