Newly developed C30 packing materials
Substrate-depending preferable combination characters

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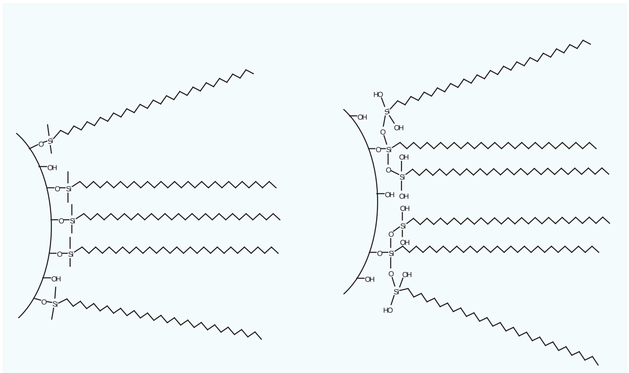
1. Introduction

The C18-modified silica gel packing materials are roughly classified in terms of three major categories of polymeric / monomeric bonding mode, endcapped / non-endcapped status and extent of coverage by carbon chains.

The polymeric / monomeric bonding mode mainly influences the planarity-cognitive capacity, the endcapped / non-endcapped status is associated with hydrogen bonding capacity and the extent of coverage by carbon chain gives differences in hydrophobicity.

Recently, C30 medium is attracted not only for separation of carotenoids but also different selectivity compared with standard type ODS medium. It also has an excellent planarity-cognitive capacity.



2. Experiments

The following 4 kinds of modified silica gel packing material were prepared starting from silica gel with the same surface area, pore size and pore volume. In each packing material, two different coverage material were synthesized by changing amount of coupling reagent.

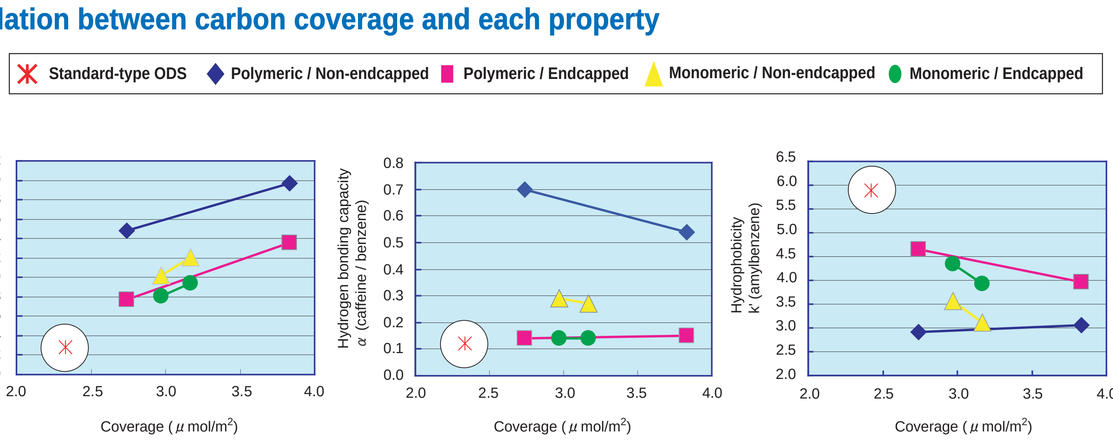
Bonding Mode	Endcapping
Polymeric	Non-endcapped
	Endcapped
Monomeric	Non-endcapped
	Endcapped

The following fundamental properties of these packing material were evaluated.

- Planarity-cognitive capacity (α (triphenylene / *o*-terphenyl))
- Hydrogen bonding capacity (α (caffeine / benzene))
- Hydrophobicity (k' (amylbenzene))

3. Results and Discussion

Correlation between carbon coverage and each property



In the polymeric materials, the planarity-cognitive capacity is getting higher with increasing carbon coverage. Especially, non-endcapped polymeric material show higher value than endcapped material.

On monomeric materials, the planarity-cognitive capacity shows similar value to endcapped polymeric material.

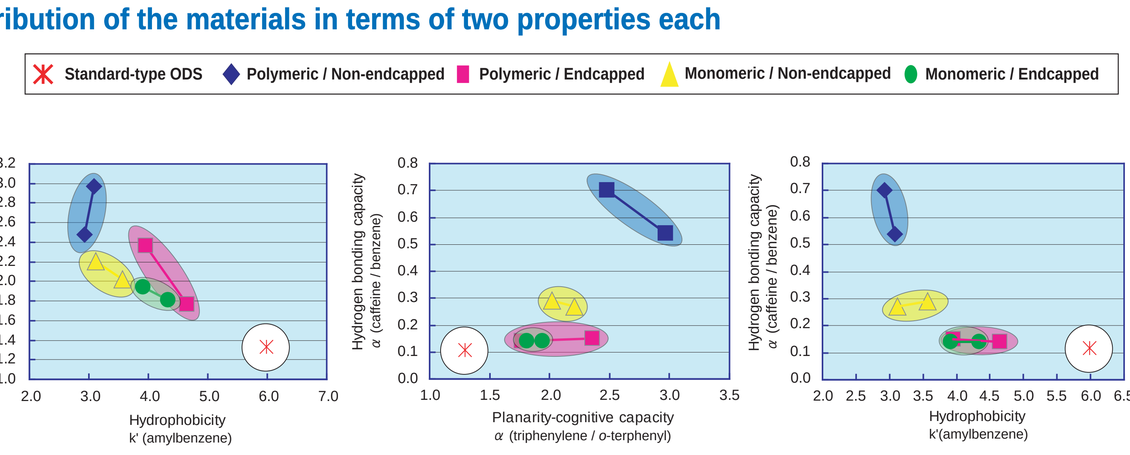
The non-endcapped polymeric material shows remarkably higher hydrogen bonding capacity than the other. The capacity decreases with increasing carbon coverage.

Endcapped materials show similar value compared with standard-type ODS.

In these 4 materials, the hydrophobicity is lower than the standard-type ODS.

Especially for non-endcapped polymeric material has lowest hydrophobicity.

Distribution of the materials in terms of two properties each

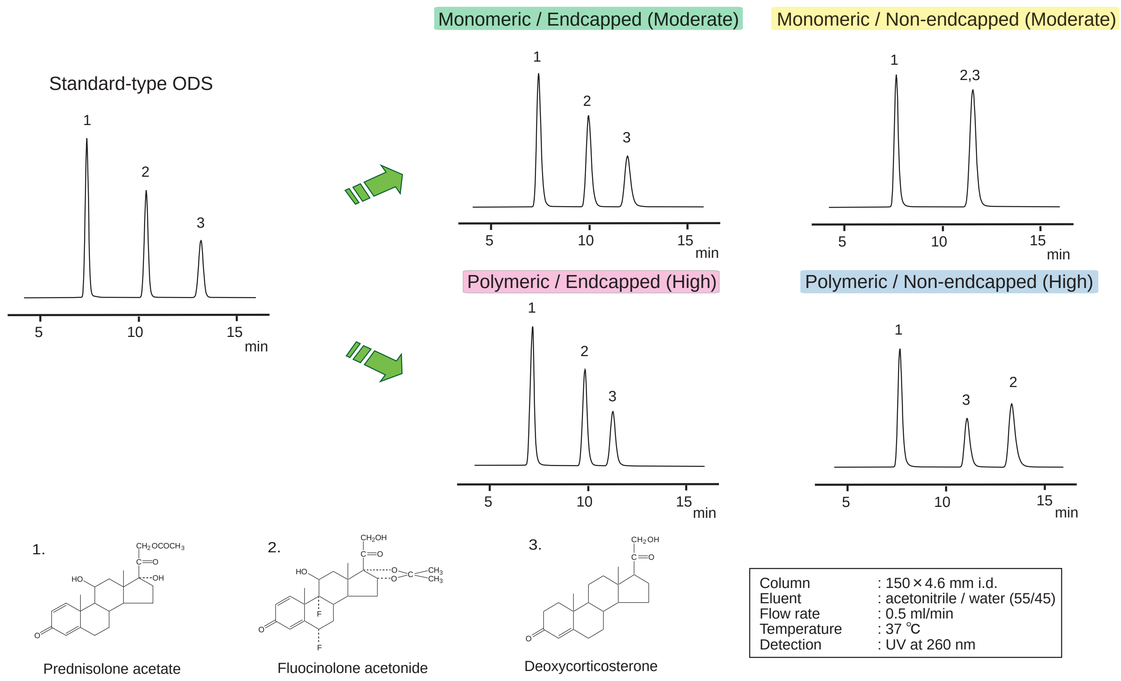


As compared with the standard-type ODS (monomeric / endcapped, marked with ＊in the figures), the materials used in this study show great differences in the fundamental separation properties. When not endcapped, the difference between the polymeric-type and monomeric-type are particularly significant. These differences suggest a possibility that some compounds hardly separable with the standard-type ODS may be separated favorably with these materials peculiar characteristics.

4. Applications

Adrenocorticosteroids

In separation of polymeric / non-endcapped material, an order of the elution of peak 2 and 3 is changed compared to standard-type ODS. In this case, the hydrogen bonding capacity greatly contributes to retention of hydrophilic compounds.

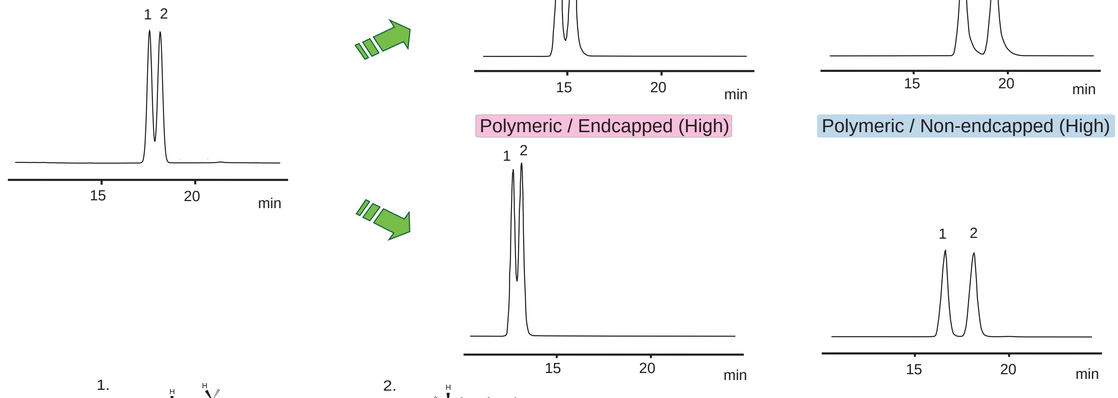


1. Prednisolone acetate
2. Fluocinolone acetonide
3. Deoxycorticosterone

Column	: 150×4.6 mm i.d.
Eluent	: acetonitrile / water (55/45)
Flow rate	: 0.5 ml/min
Temperature	: 37 ℃
Detection	: UV at 260 nm

Vitamin D_2 and Vitamin D_3

The non-endcapped materials can separate Vitamin D_2 and Vitamin D_3 which are hardly separable with the standard-type ODS. Especially, the polymeric / non-endcapped material shows excellent separation. In this case, not only planarity-cognitive capacity but also hydrogen bonding capacity seem to play an important role in separation.

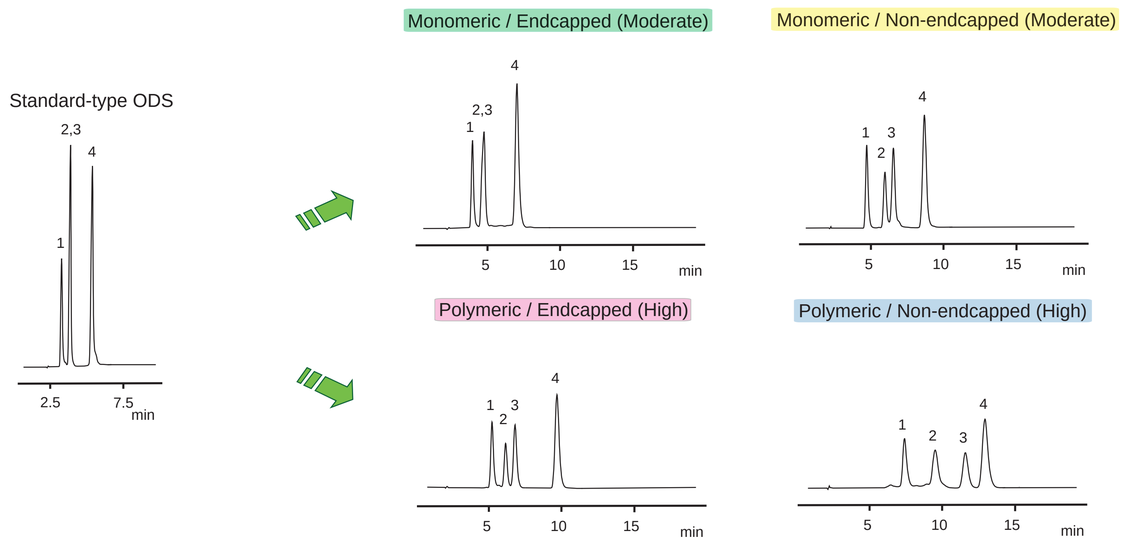


1. Ergocalciferol (Vitamin D_2)
2. Cholecalciferol (Vitamin D_3)

Column	: 150×4.6 mm i.d.
Eluent	: acetonitrile / water (95/5)
Flow rate	: 1.0 ml/min
Temperature	: 40 ℃
Detection	: UV at 265 nm

Carotenoids

C30 materials except monomeric / endcapped type show better separation of peaks 2 and 3 than standard-type ODS. Especially polymeric / non-endcapped material shows excellent selectivity due to high hydrogen bonding capacity and high planarity-cognitive capacity.



1. Astaxanthin
2. Lutein
3. Zeaxanthin
4. Canthaxanthin

Column	: 150×4.6 mm i.d.
Eluent	: methanol / *t*-butylmethylether / water (81/15/4)
Flow rate	: 1.0 ml/min
Temperature	: 30 ℃
Detection	: VIS at 480 nm

5. Conclusions

In this study, various combination of the polymeric / monomeric, endcapped / non-endcapped and two different coverage characters were evaluated systematically. As a result, we obtained the packing materials having greatly different properties as compared with the standard-type ODS. For examples, high planarity-cognitive and high hydrogen bonding capacity are unique characters for C30 materials. These characters enhance recognition ability for subtle structure differences such as structural isomers, vitamins, steroids and carotenoids.

As we mentioned, C30 materials are applied to separate of similar structure compounds. Particularly, polymeric / non-endcapped material shows excellent selectivity and it is expected to play a special role in HPLC.