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Identification of Elucamide as an Organic Compound which Affects Mechanical Properties of Poly (vinyl chloride) - Inorganic Filler Binary Composite System

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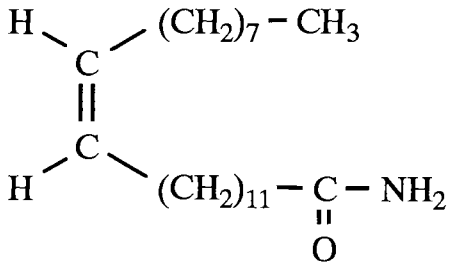
The mechanical properties (yield strength) were measured for poly (vinyl chloride) - inorganic compound binary composite systems. Among them, only the PVC-talc system did not show the decreasing yield strength with increasing talc content. Elucamide was isolated from talc and identified as an organic compound which can provide inhibitory action of such a decreasing yield strength.

Recently, much importance has been attached to highly efficient plastics including inorganic fillers.[1-3] For example, talc and mica have been added to plastics for the purpose of enhancing stiffness.[4-6] We already reported that the mechanical property (yield strength) for the PVC-talc binary composite system does not decrease up to 50 phr (per hundred resin) talc content, while that for the PVC-$CaCO_3$ binary system does decrease remarkably.[1] We also observed using SEM (after the tensile break process) that certain fibrous materials seem to interconnect between organic PVC and inorganic talc.[1]

Other inorganic fillers such as mica and kaolin show the decreasing behavior of yield strength similarly to that observed for the PVC-$CaCO_3$ system, as shown in Fig. 1. Therefore, the specificity of the PVC-talc system has now become evident. We describe here the identification of elucamide **1** (cis-13-docosenamide) which is contained in talc as one of the organic compounds providing inhibitory action for such a decreasing yield strength.



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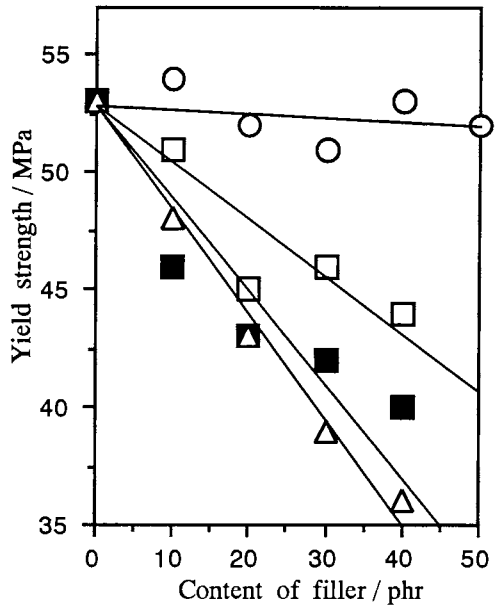


Fig.1. Yield strength of PVC compounds.
(○:Talc, □:Mica, ■ :Kaolin, △ :$CaCO_3$)

Commercial talc (1.5 kg) [7] was treated by Soxhlet extraction with dichloromethane for 160 h. After evaporation of the solvent, 150 mg (0.01 wt%) of extract was taken. HPLC separation was undertaken using an ordered phase column (Lichrosorb Si60), an RI detector, and an eluent of ethyl acetate : hexane (1:1) (Fig. 2). A fraction of the longest retention time (12.9 min) (E peak) was collected. The extract showed a single peak by GLC determination (column material ; Shimadzu SE30, detector ; FID) (1.6 mg of the isolated compound from 12.3 mg of the extract). After several trials, the compound was identified as elucamide **1** on the basis of ^{1}H-NMR, ^{13}C-NMR, EI-MS, FT-IR spectral data, and melting point.[8] Further, the compound was confirmed by referring to commercial sample of **1** to be an authentic material.

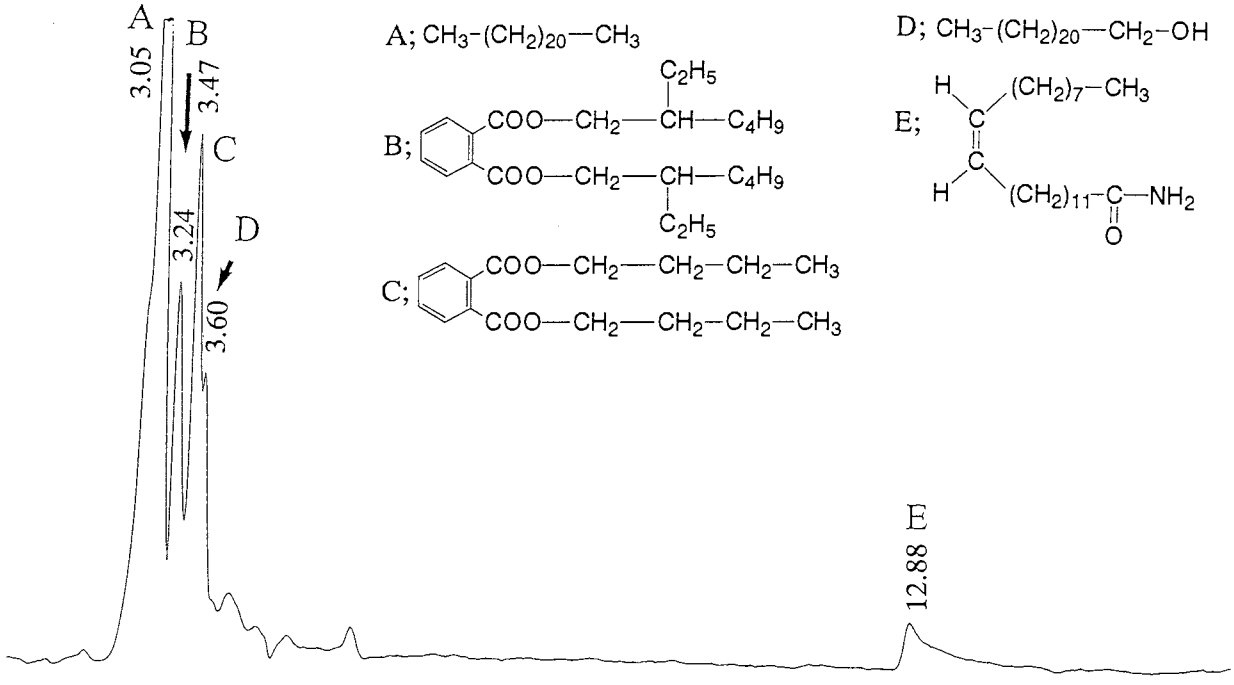


Fig.2. High performance liquid chromatogram.

The other compounds for peaks appearing in Fig. 2 were also isolated and identified as n-docosane [$CH_3(CH_2)_{20}CH_3$; A peak], di-2-ethylhexyl phthalate [$C_6H_4\{COOCH_2CH(C_2H_5)C_4H_9\}_2$; B peak], dibutyl phthalate [$C_6H_4\{COO(CH_2)_3CH_3\}_2$; C peak], and 1-docosanol [$CH_3(CH_2)_{21}OH$; D peak].[9)]

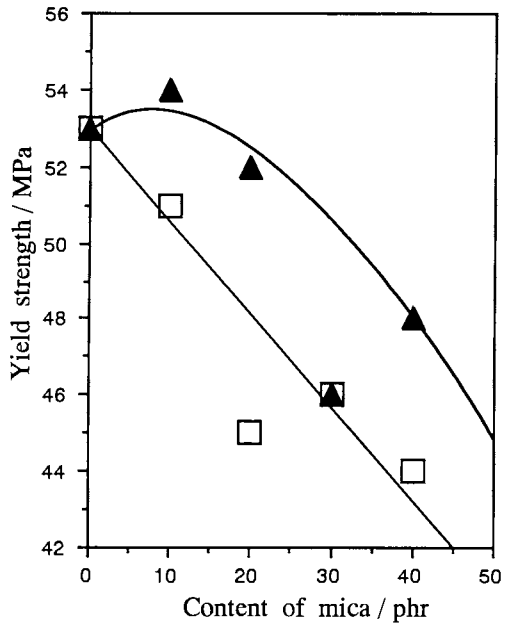
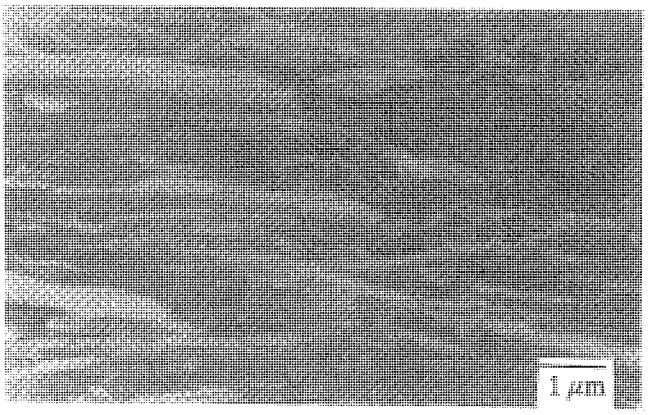


Fig.3. Yield strength of PVC compounds.
(□: Mica, ▲: Mica-Erucamide (0.5 phr))



(Mica 20 phr + Erucamide, 0 phr)

(Mica 20 phr + Erucamide, 0.5 phr)

Fig.4. Electron micrograph of test piece after breaking.

As a next step, we chose the PVC-mica binary system which shows the decreasing yield strength with increasing mica content, and we examined whether **1** actually exhibits an inhibitor action or not in this system. As is shown in Fig.3, effective inhibitor actions have been demonstrated. Moreover, newly developed fibrous materials interconnecting PVC with mica have been observed using SEM (Fig. 4). Therefore, more generally, we tested the effectiveness of the addition of another aliphatic amide with a long alkyl chain such as behenamide ($C_{21}H_{43}CONH_2$) or lauramide ($C_{11}H_{23}CONH_2$) to a PVC-talc binary system. Such an amide can successfully provide a high degree of the increasing yield strength, as shown in Fig.5.

In summary, we have found that elucamide **1** and related compounds are promising organic compounds which can strengthen the mechanical property such as the yield strength of organic PVC-inorganic filler binary composite systems. We are studying such additive effects further.

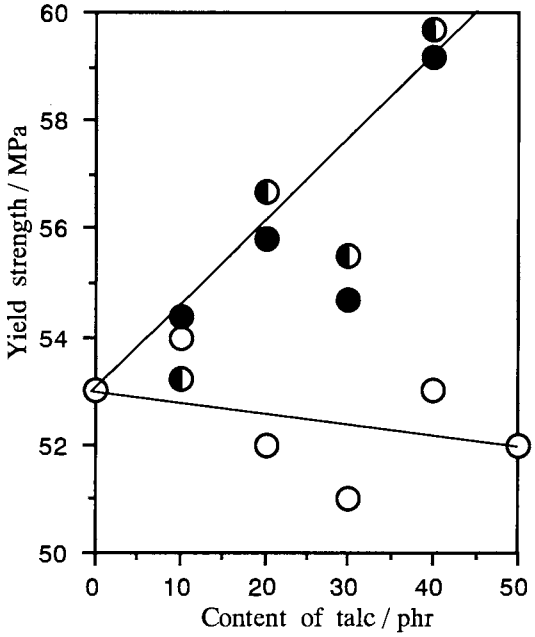


Fig.5. Yield strength of PVC compounds.
(○:Untreated, ●:Talc-Lauramide (0.5 phr), ◐:Talc-Behenamide (0.5 phr))

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7) Fuji Talc Corp. NK-#64.

8) Colorless powder: ^{1}H-NMR(360 MHz, $CDCl_3$) δ 5.35(m, 2H), 2.20(t, 2H), 2.02(m, 4H), 1.61(m, 2H), 1.26 (m, 28H), 0.88(m, 3H); ^{13}C-NMR(90.56 MHz, $CDCl_3$) δ 175.5, 129.9, 35.9, 31.9, 29.8-29.3, 27.2, 25.5, 22.7, 14.0; MS(70 eV) m/e 337(M+); FT-IR (KBr, cm^{-1}) 3400, 3180, 2950, 1650, 1470, 1420; mp 82 °C.

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9) We have not investigated mechanical properties of these materials. At first, we regarded that **1** is the most promising material from the standpoint of the possibility of chemical reaction or adhesion with PVC.

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