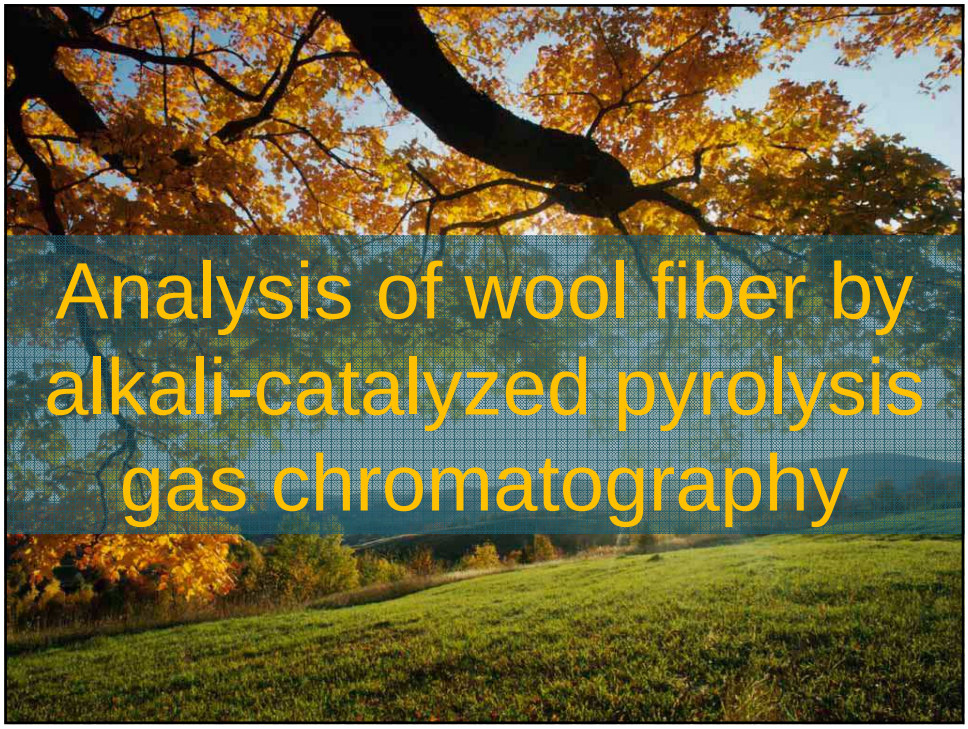
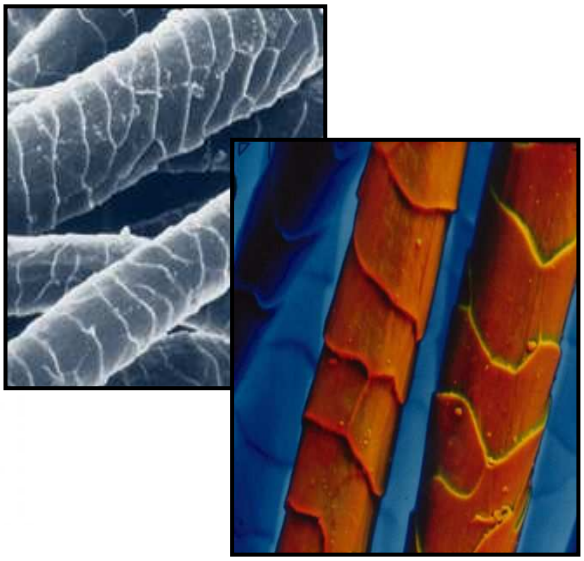
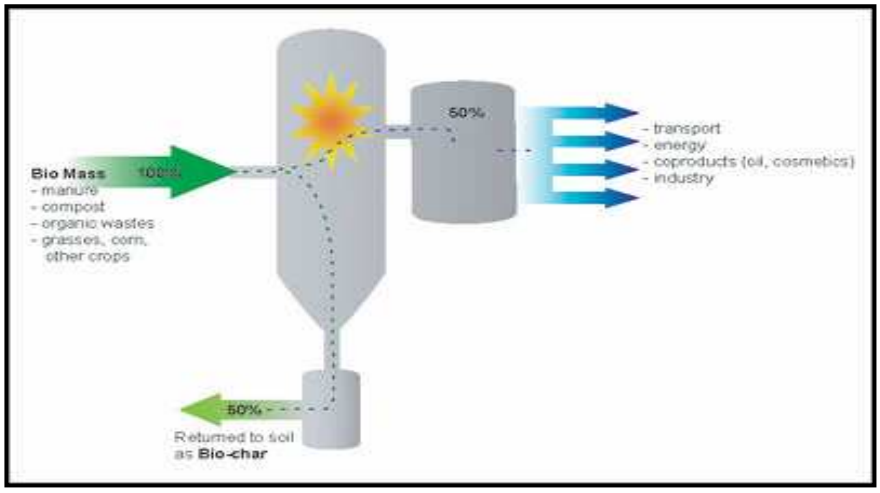

Analysis of wool fiber by
alkali-catalyzed pyrolysis
gas chromatography


WOOL

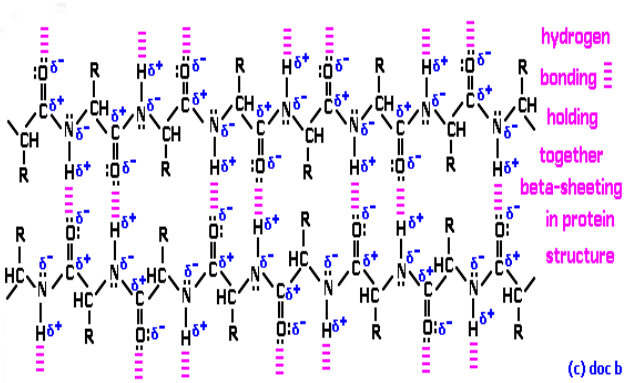
WOOL

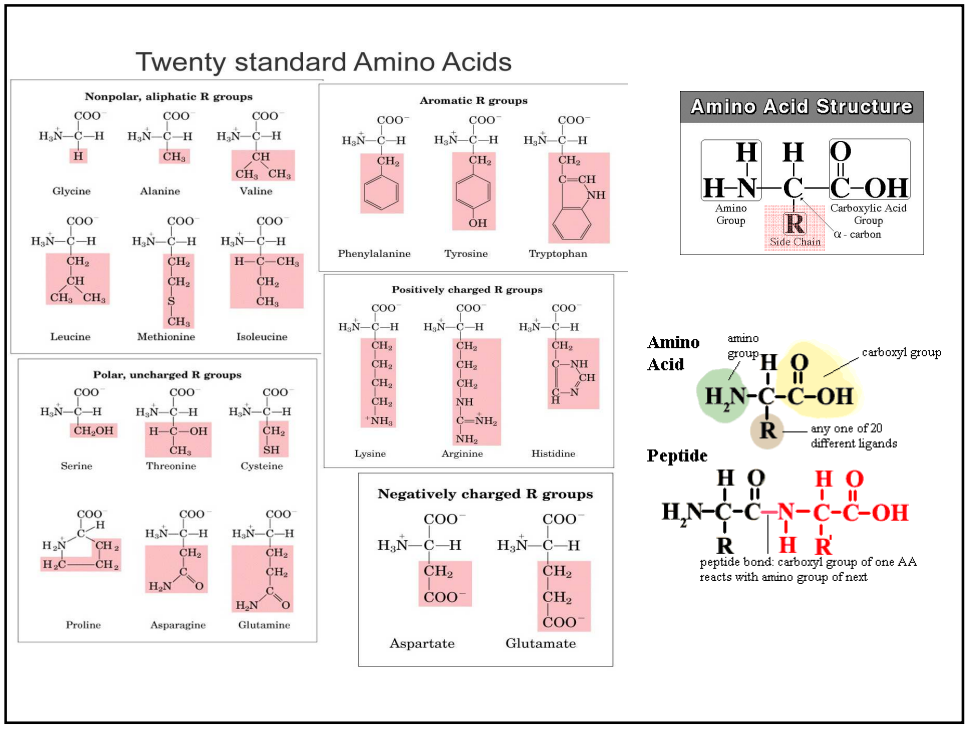


Pyrolysis Process



WOOL chemical



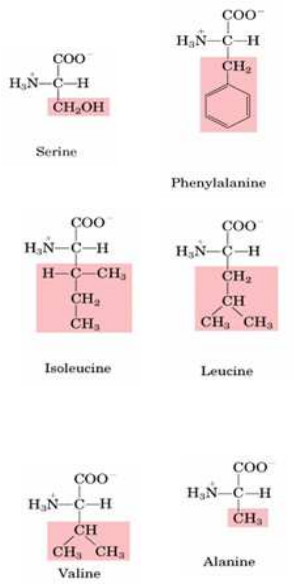


Procedures for alkali-catalyzed PyGC

A sample (approx. 20 ug) consisting of four single wool fibers each approx. 10 mm in length was placed on a piece of pyrofoil.

After adding 0.5 ul of 25% aqueous sodiumhydroxide, the sample was wrapped in pyrofoil for use in PyGC.

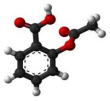
Chemical reaction



Pyrolysis

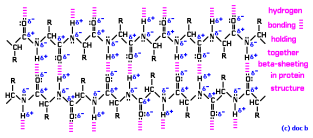
25% NaOH

Volatile molecule

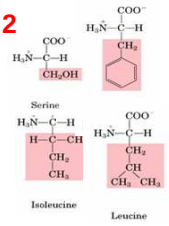


Pyrolysis

1

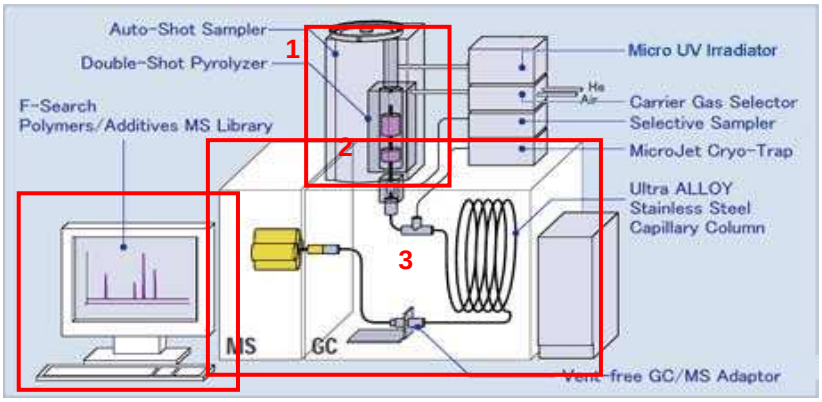


2



3

Volatile molecule



PyGC-MS conditions

Pyrolyzer	
Instrument	Japan analytical industry Curie point Model JHP-2 (GC), JHP-3 (GC-MS)
Pyrolysis	590°C; 3 s
Oven temperature	120°C
Pipe temperature	250°C
GC	
Instrument	Shimadzu GC-7AG
Detector	FID
Column	J&W DB-5 (0.25 mm i.d.×30 m), film thickness 0.25 μm
Column temperature	60°C (8 min)–230°C (10°C/min)
Injection temperature	230°C
Detector temperature	230°C
Carrier gas	Nitrogen, flow rate 1.0 ml/min, split 30:1
Make up gas	Nitrogen, flow rate 50 ml/min
GC-MS	
Instrument	Shimadzu QP-1000
Column	J&W DB-5 (0.25 mm i.d.×30 m), film thickness 0.25 μm
Column temperature	60°C (8 min)–230°C, 10°C/min
Injection temperature	230°C
Carrier gas	Helium, flow rate 1.0 ml/min
Make up gas	Helium, flow rate 40 ml/min
Ion source energy	70 eV(EI), 20 eV(CI)
Reaction gas	Isobutane

Results

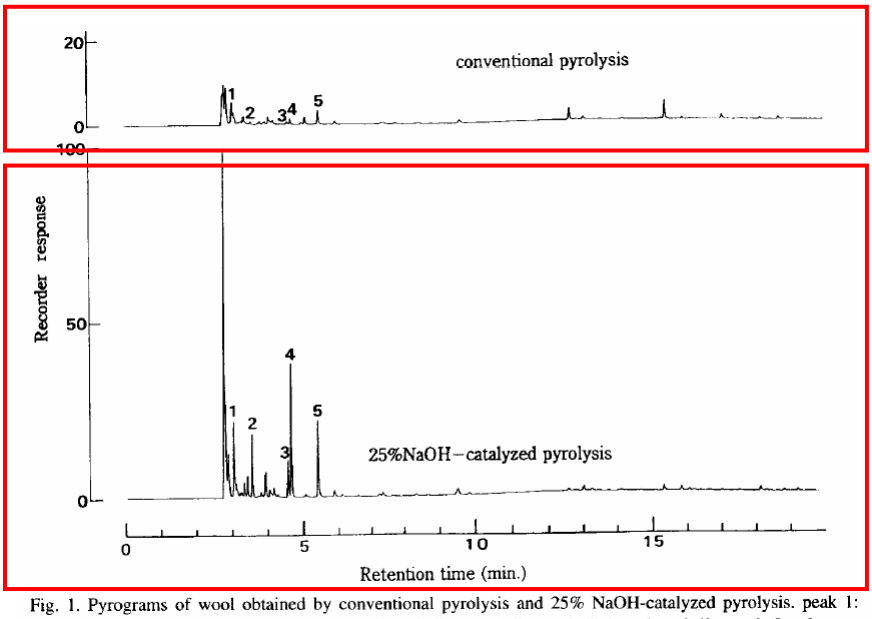


Fig. 1. Pyrograms of wool obtained by conventional pyrolysis and 25% NaOH-catalyzed pyrolysis. peak 1: acetonitrile; peak 2: isobutyronirile; peak 3: 2-methylbutyronitrile; peak 4: isovaleronitrile; peak 5: toluene.

MS

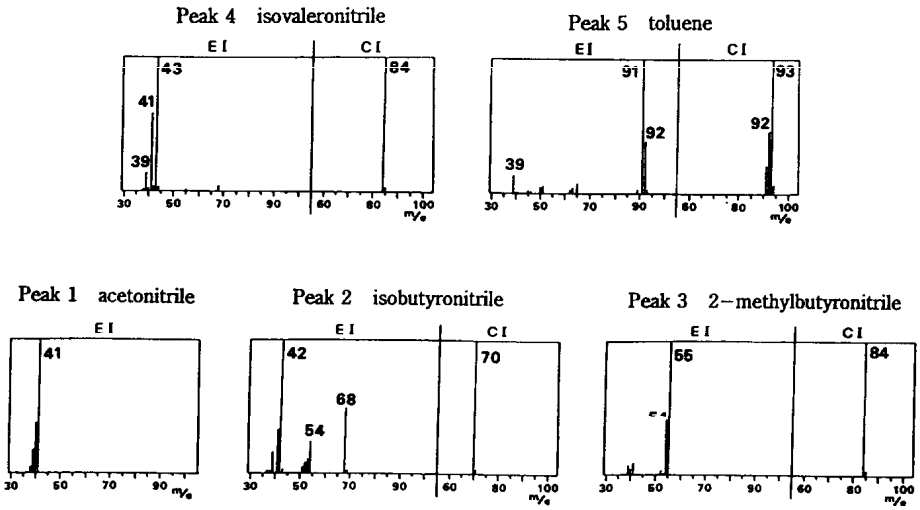


Fig. 2. Mass spectra of major degradation products in alkali-catalyzed pyrolysis of wool using EI and CI ionization.

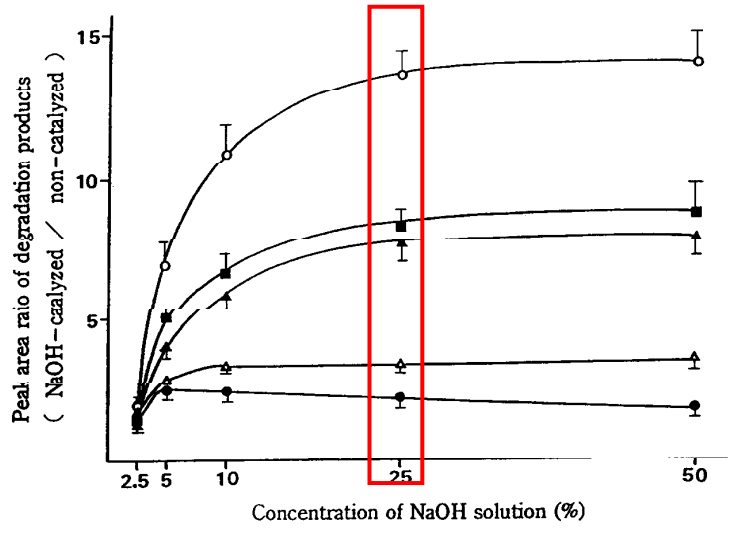
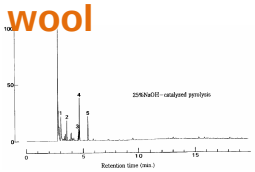


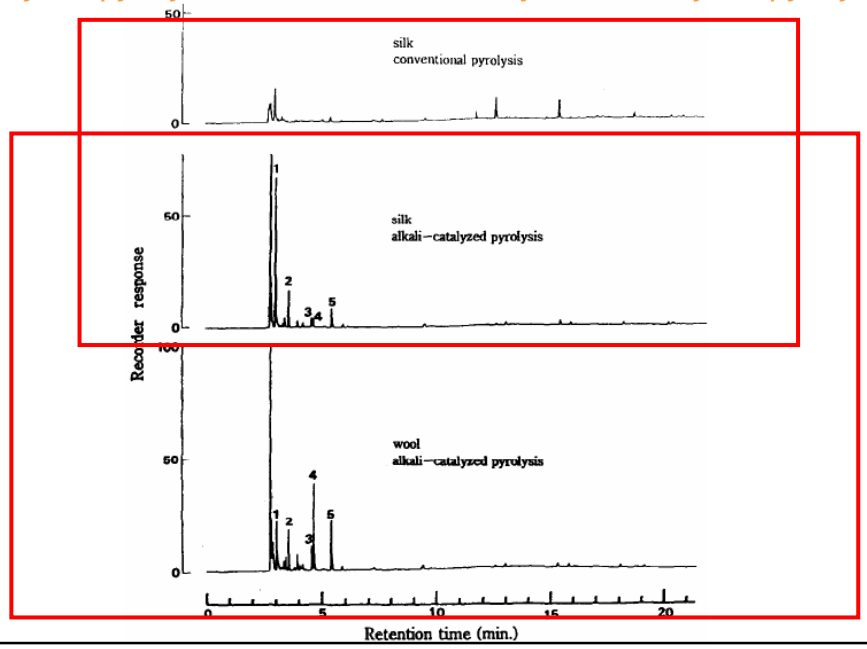
Fig. 3. Effect of concentration of NaOH solution on yields of degradation products in wool pyrolysis. Data presented: mean±S.D. of five samples. ●: acetonitrile; ▲: isobutyronitrile; ■: 2-methylbutyronitrile; ○: isovaleronitrile; △: toluene.

Degradation products identified on the alkali-catalyzed pyrolysis of wool

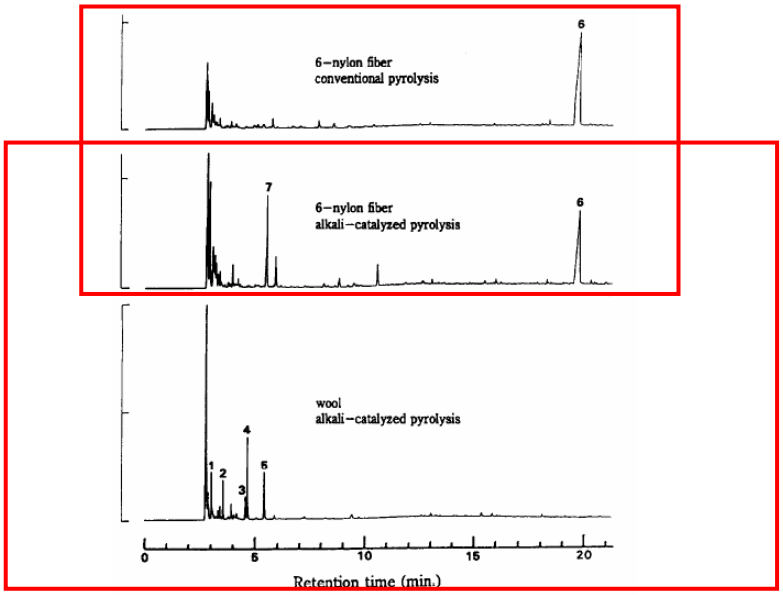


Peak No. [a]	Degradation products	Amino acid residues assigned
1	CH_3-CN (Acetonitrile)	NH- CH_3-CH-CO- (Alanine)  CO- (Proline)
2	CH_3-CH(CH_3)-CN (Isobutyronitrile)	NH- CH_3-CH(CH_3)-CH-CO- (Valine)
3	CH_3-CH_2-CH(CH_3)-CN (2-Methylbutyronitrile)	NH- CH_3-CH_2-CH(CH_3)-CH-CO- (Isoleucine)
4	CH_3-CH(CH_3)-CH_2-CN (Isovaleronitrile)	NH- CH_3-CH(CH_3)-CH_2-CH-CO- (Leucine)
5	 CH_3 (Toluene)	NH-  CH_2-CH-CO- (Phenylalanine)

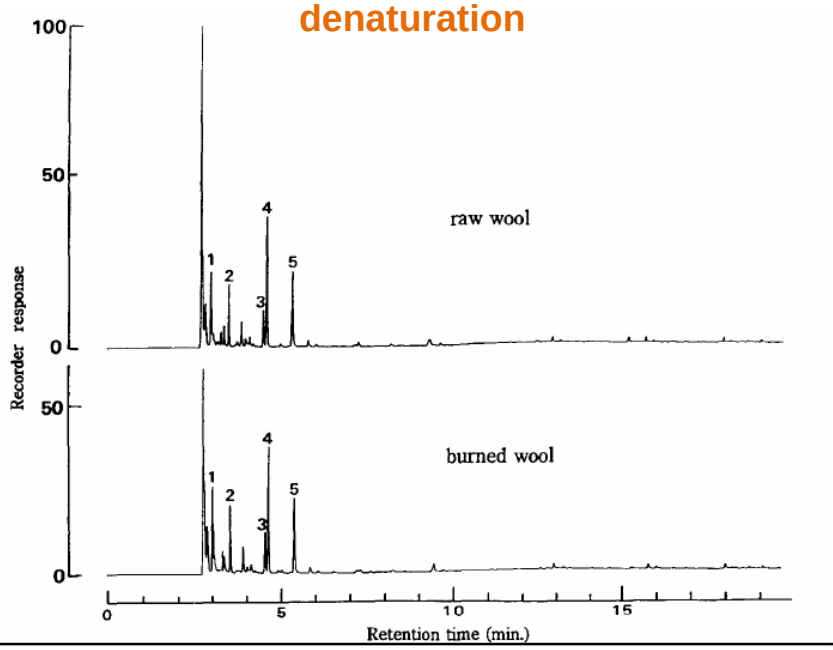
Pyrograms of silk obtained by conventional pyrolysis and alkali-catalyzed pyrolysis and wool obtained by alkali-catalyzed pyrolysis.



Pyrograms of 6-nylon fiber obtained by conventional pyrolysis and alkali-catalyzed pyrolysis and wool obtained by alkali-catalyzed pyrolysis.



Analysis of wool fiber subjected to thermal denaturation



Conclution

Question