



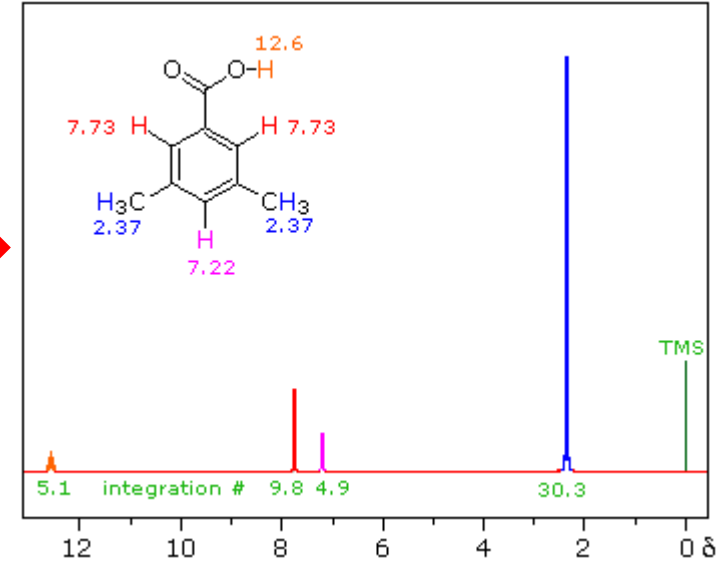
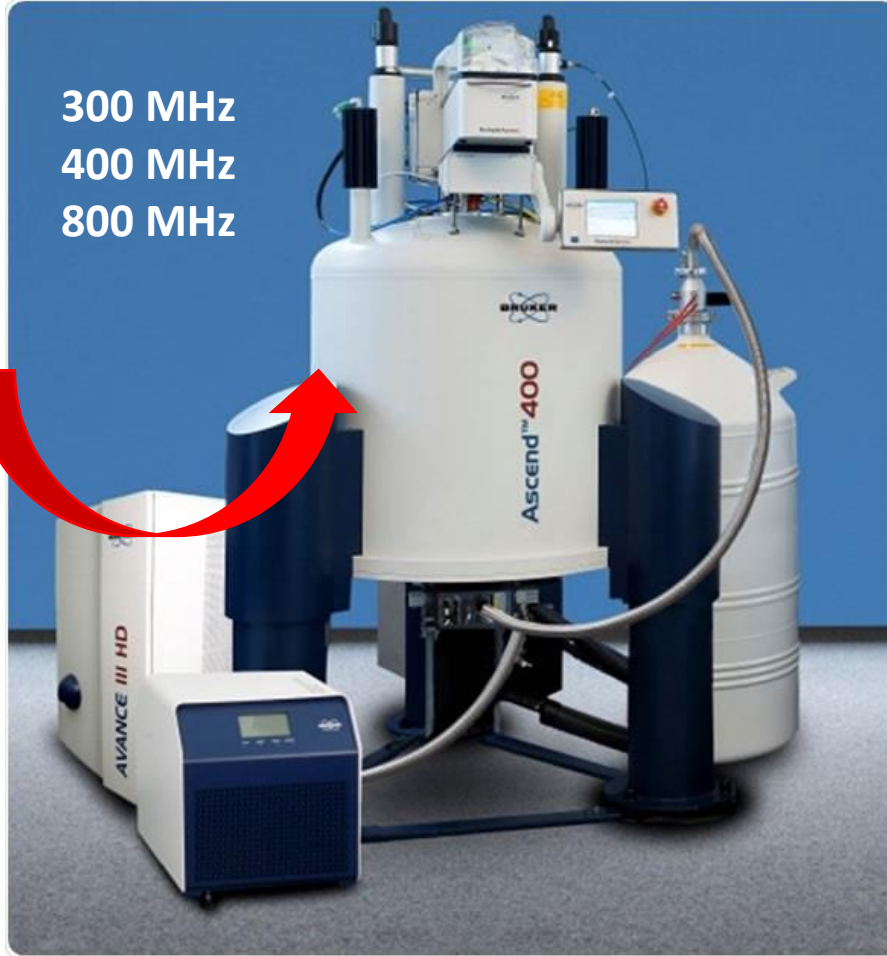
ORTA DOĞU TEKNİK ÜNİVERSİTESİ  
MIDDLE EAST TECHNICAL UNIVERSITY

## Gıda Bilimi ve Mühendisliğinde Düşük Alanlı NMR Relaksometre ve Manyetik Rezonans Görüntüleme Tekniklerinin Kullanımı

Doç. Dr. Mecit Halil Öztop  
Orta Doğu Teknik Üniversitesi  
Gıda Mühendisliği Bölümü



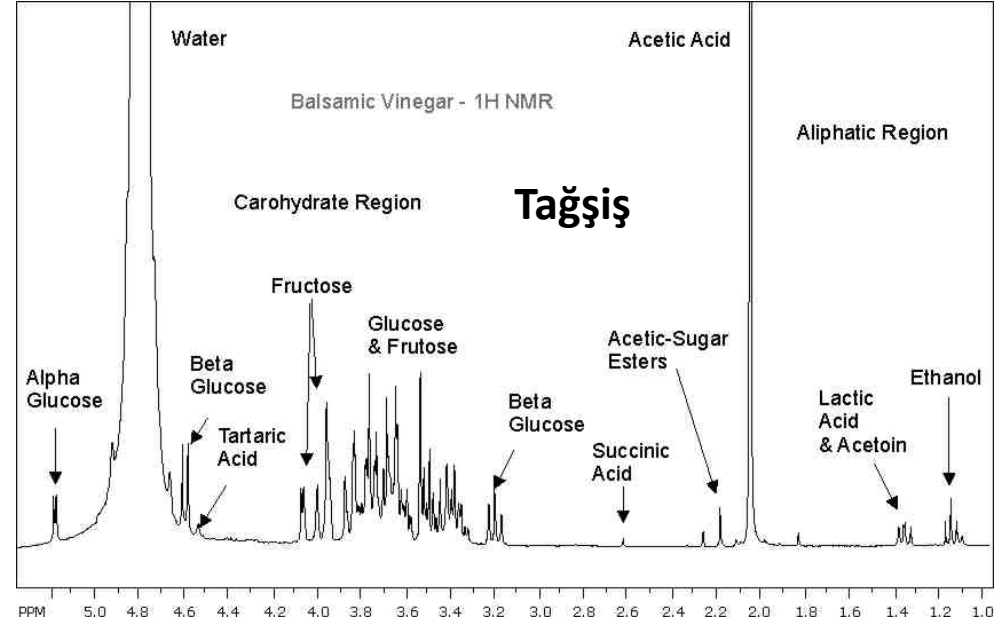
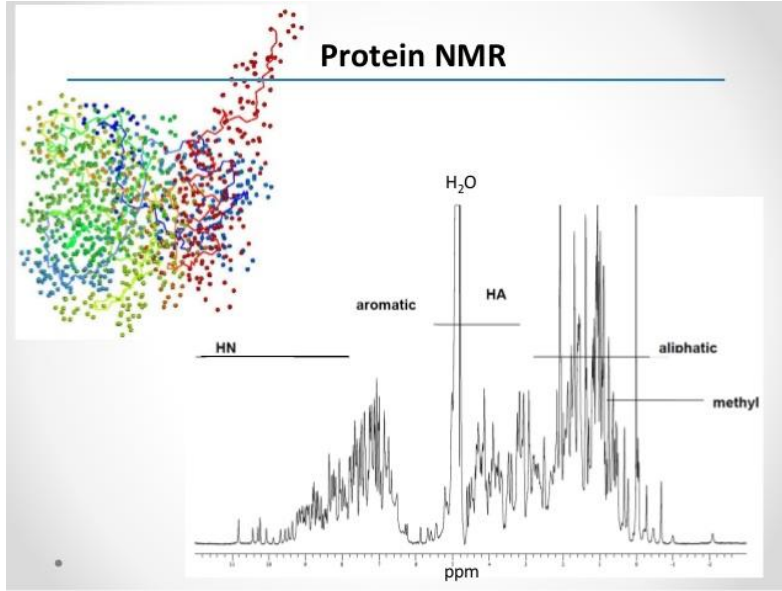
# Nükleer Manyetik Rezonans ve (NMR) ve akla gelenler ????



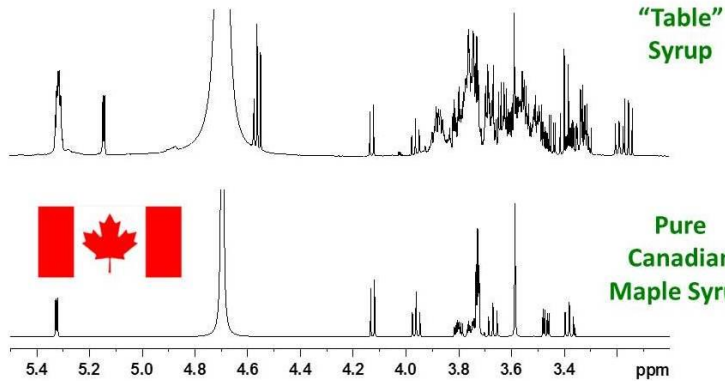
- Spektroskopi ?
- Food-Omics
- Metabolomics
- Yüksek homojenite
- Yüksek rezolüsyon

3-5 mm numune hacmi

# Nükleer Manyetik Rezonans Spektroskopisi

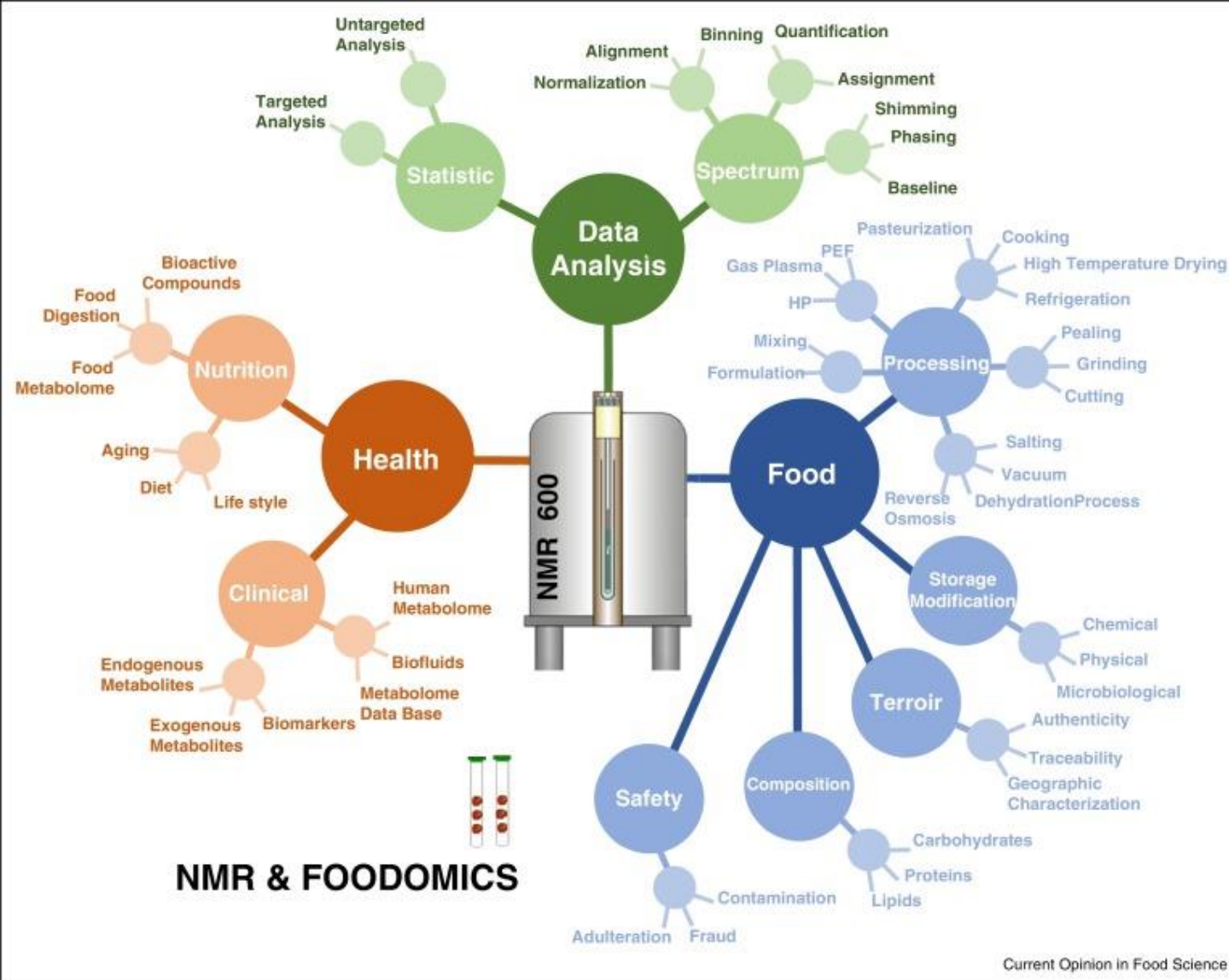


600 MHz <sup>1</sup>H NMR Spectra of "Maple Syrup"



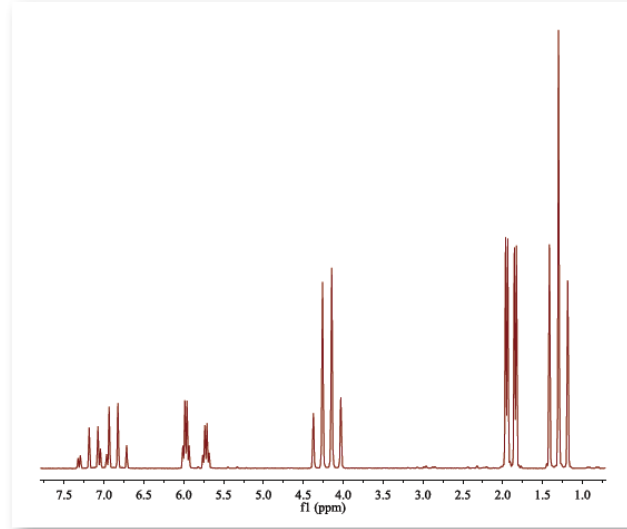
Veri işleme ??

Yorumlama ??

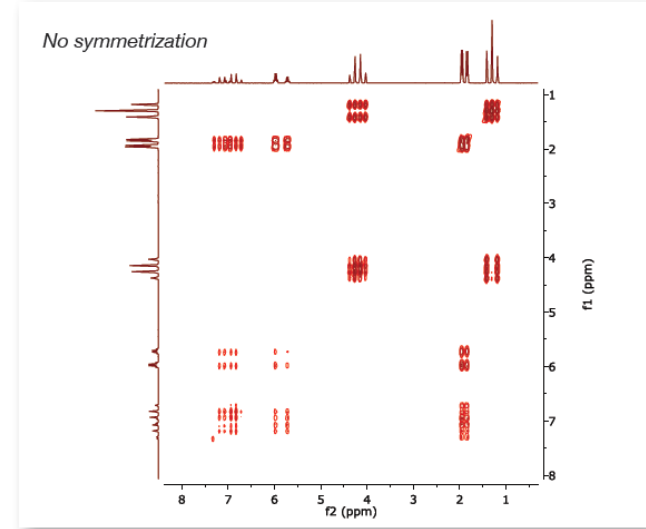


Alessia Trimigno, Flaminia Cesare Marincola , Nicolò Dellarosa , Gianfranco Picone , Luca Laghi, 2015. Definition of food quality by NMR-based foodomics, *Current Opinion in Food Science*, 4: 99-104

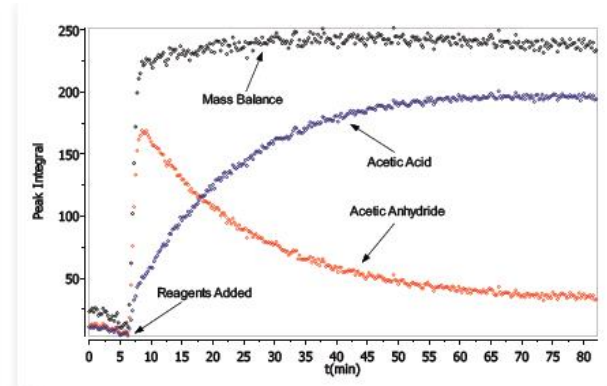
# Benç Tipi Nükleer Manyetik Rezonans Spektroskopisi



Proton spectrum of 1 M Ethyl Crotonate (Spinsolve 60, 1 minute)



COSY spectrum of 1 M Ethyl Crotonate (Spinsolve 60, 38 minutes)



Hydrolysis of acetic anhydride with mass balance  
(see Magritek Application Note 9)

# NMR

- Nükleer Manyetizma
- Nükleer Manyetizma her atomda görülmez; atomun belli bazı özellikleri nükleer manyetizmanın oluşup oluşmayacağını belirler.
- NMR aktif atomlar

Atom Numarası (Z)	Nötron Numarası(N)	Kütle Numarası (A)	Nükleer Manyetizma
çift	çift	çift	(-)
çift	tek	tek	evet
tek	çift	tek	evet
tek	tek	çift	evet

**Tablo:** Proton ve nötron sayılarının çift sayıda olmaları halinde nükleer manyetizma (-) olmaktadır. Proton veya nötron sayıları tek olduğunda ise nükleer manyetizma (+) olmaktadır.

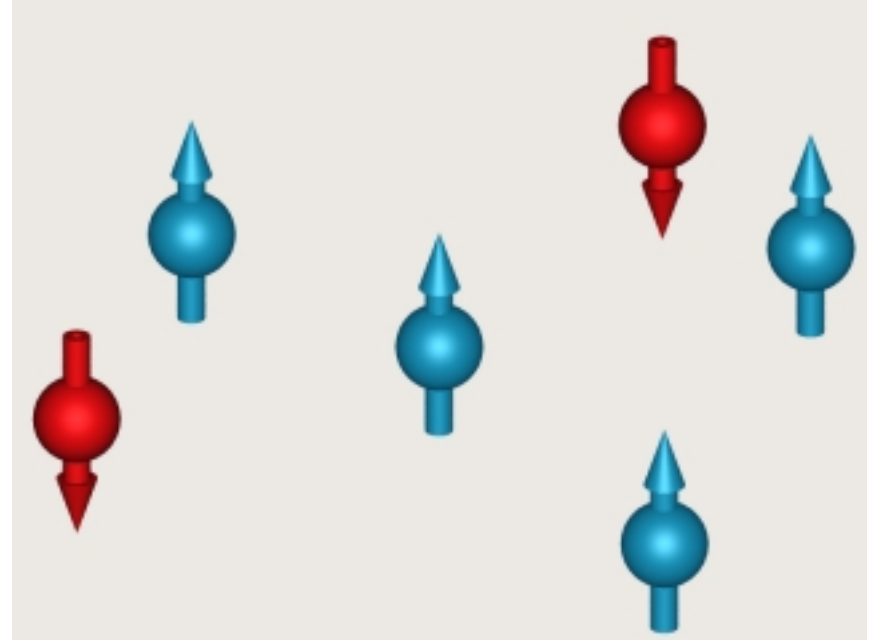
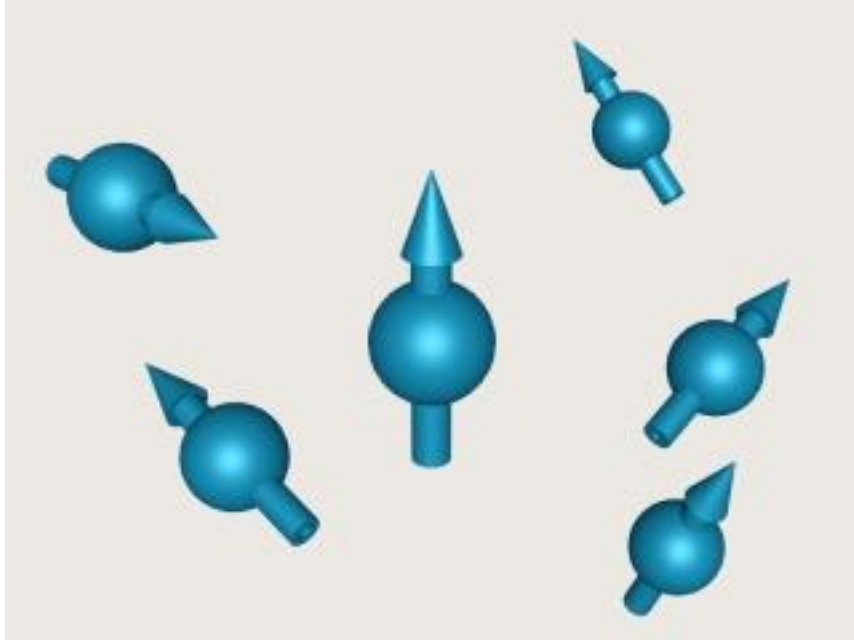


# NMR Aktif Atomlar

N	Z	Izotop	Element	% Abundance
1	0	H1	Hidrojen	99,98
1	1	H2	Hidrojen	0,02
6	7	C13	Karbon	1,10
7	7	N14	Nitrojen	99,62
7	8	N15	Nitrojen	0,38
8	9	O17	Oksijen	0,039
9	10	F19	Florin	100,0
11	12	Na23	Sodyum	100,0
12	13	Mg25	Magnesium	11,50
15	16	P31	Fosfor	100,0
17	18	C135	Klor	75,40
17	20	C137	Klor	24,60
19	20	K39	Potasyum	93,38

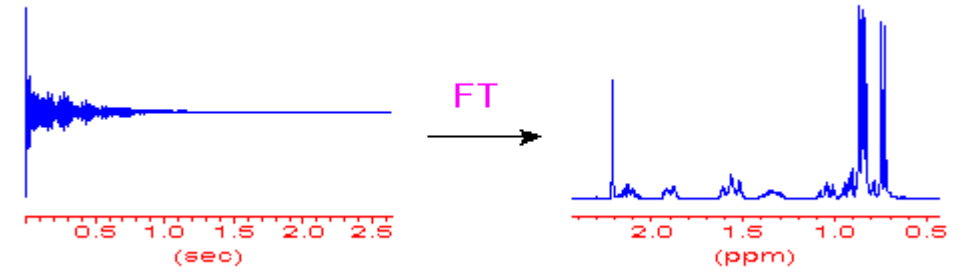
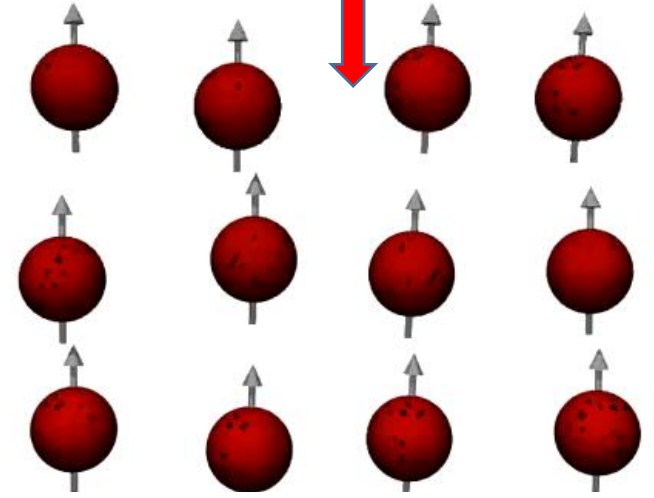
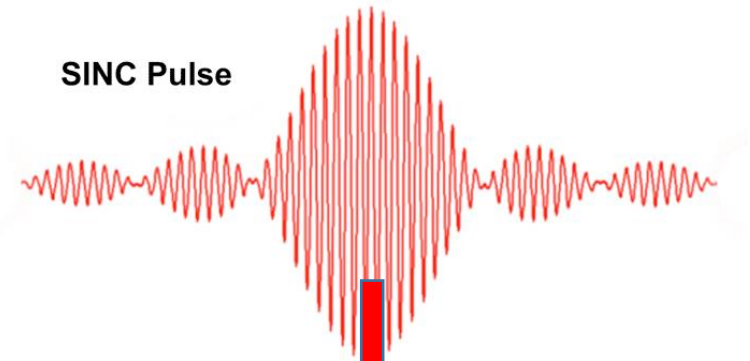
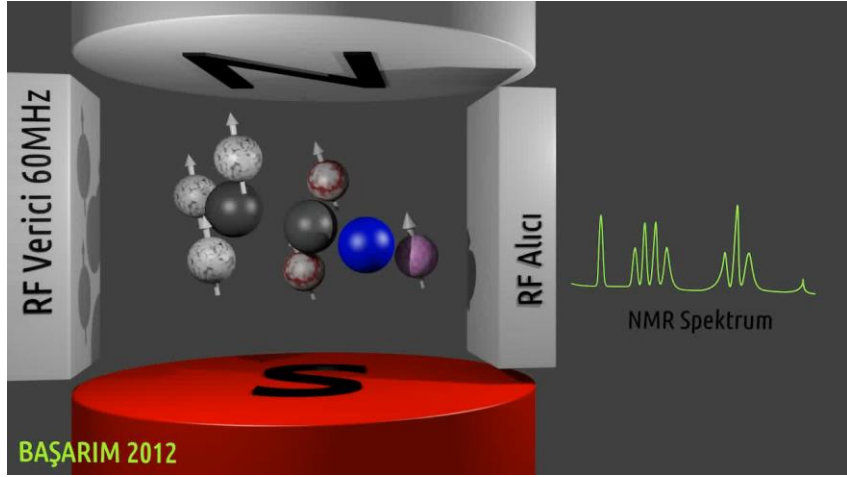
Tablo: Atom numaraları aynı, kütle numaraları farklı olan atomlara izotoplar denmektedir. Örneğin hidrojenin izotoplarından H1'de 1 proton, H2'de 1 proton ve 1 Nötron vardır.

# Spinler

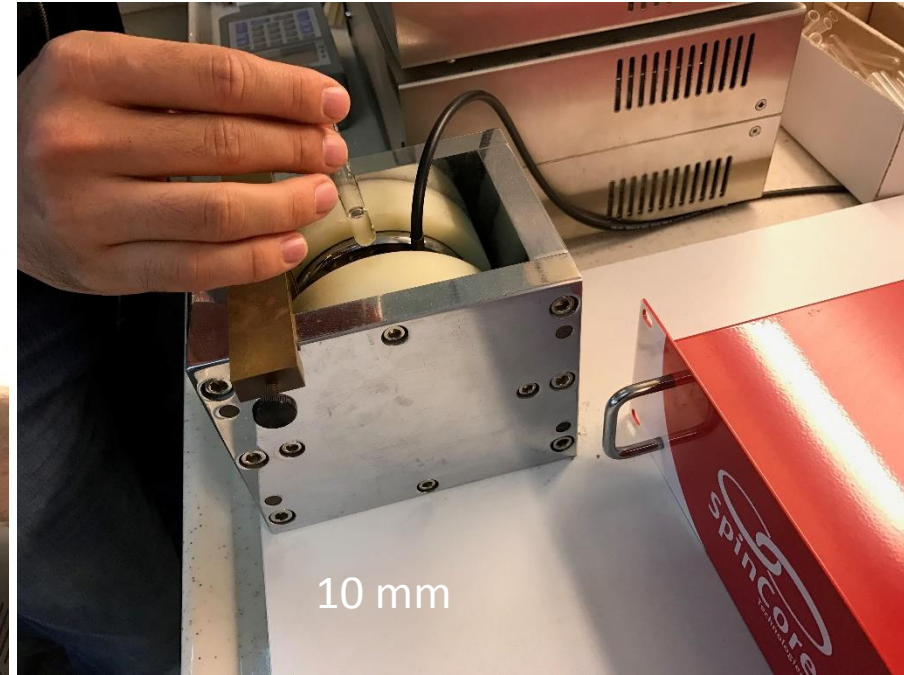
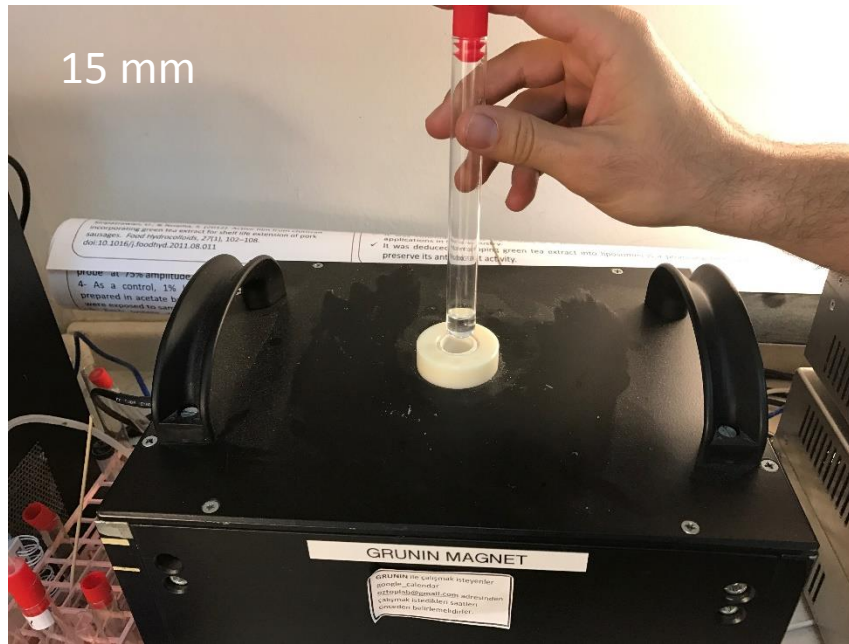




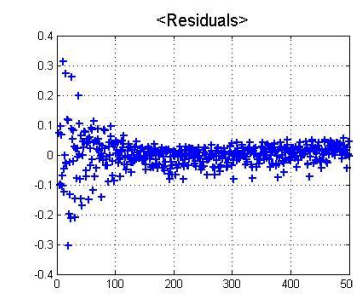
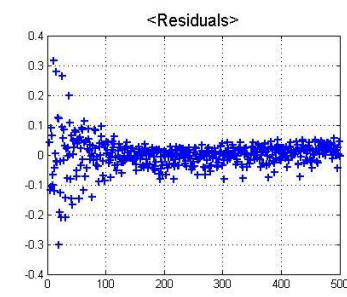
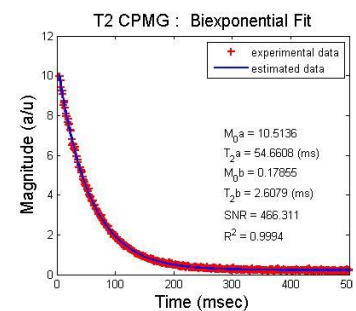
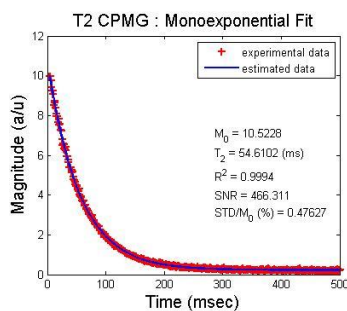
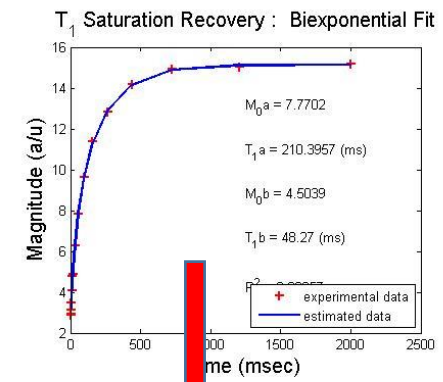
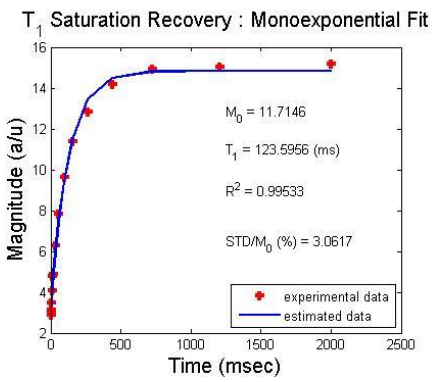
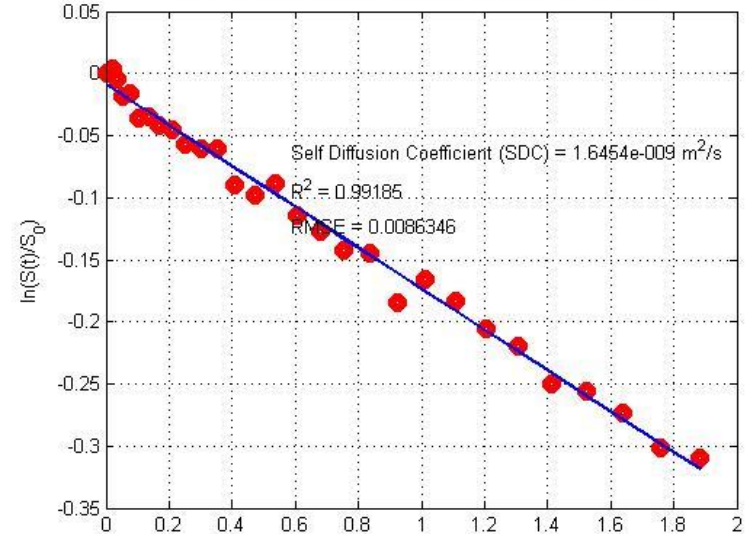
# NMR Sinyali



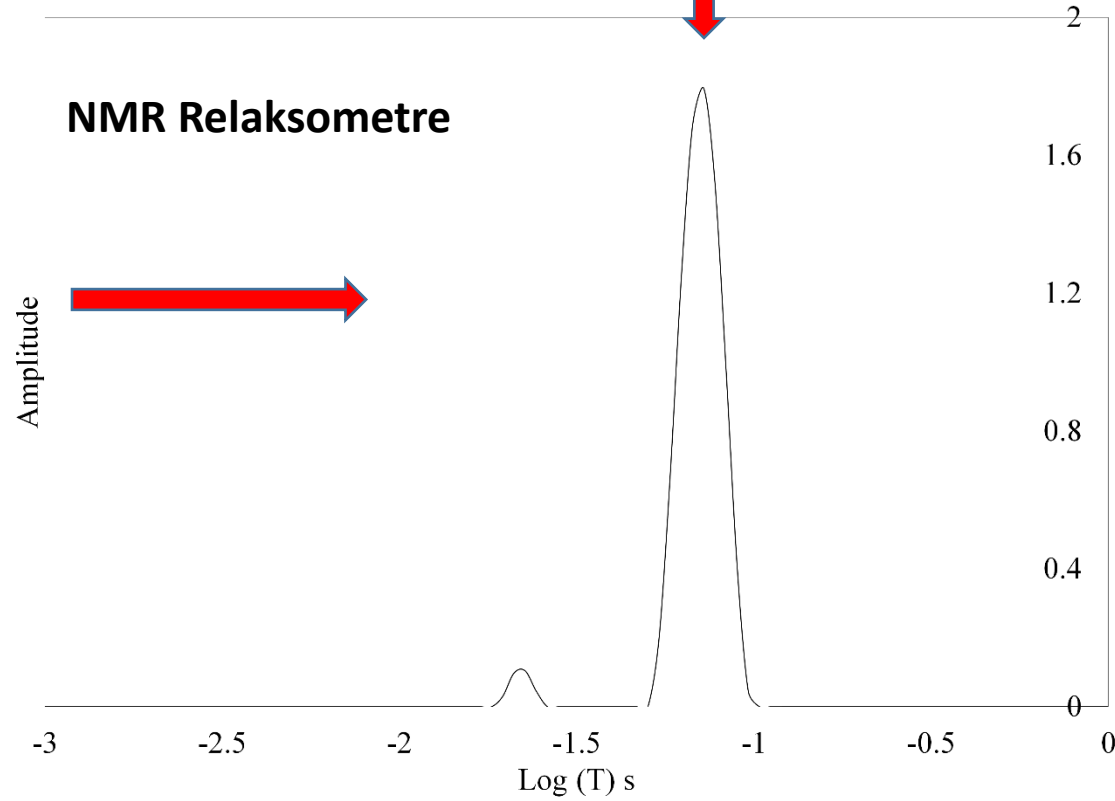
# Zamansal Alanda NMR (TD-NMR)



Self Diffusion Coefficient : Pulse Gradient Stimulated Echo



## NMR Relaksometre



# TD-NMR : Katı Yağ Analizi

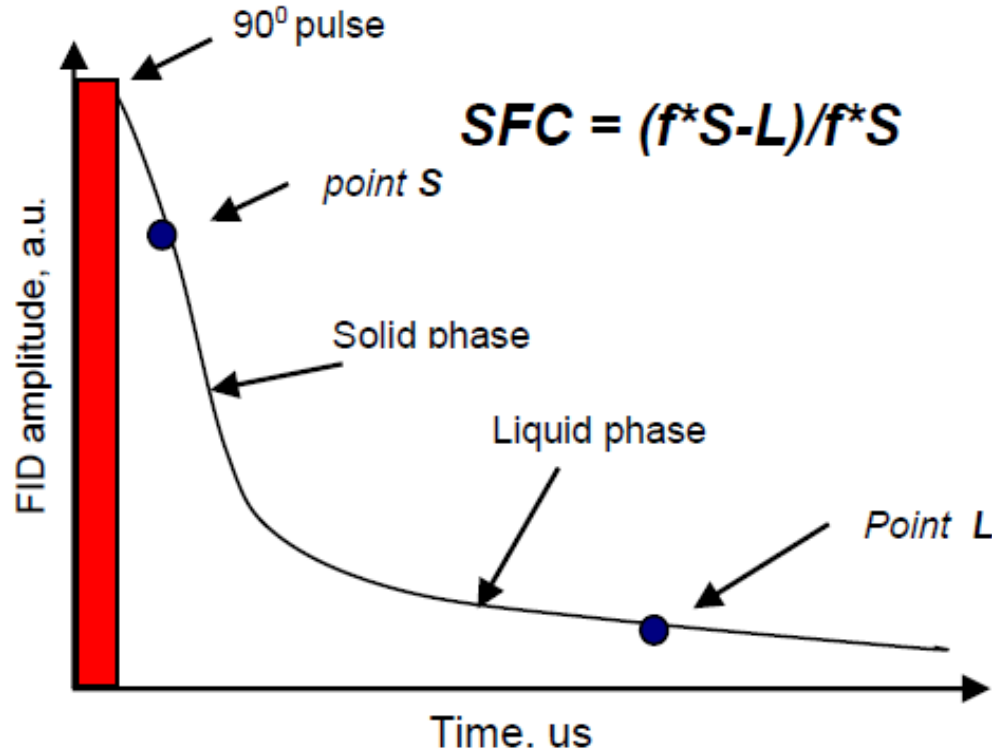


Fig. 1. SFC calculation based on FID

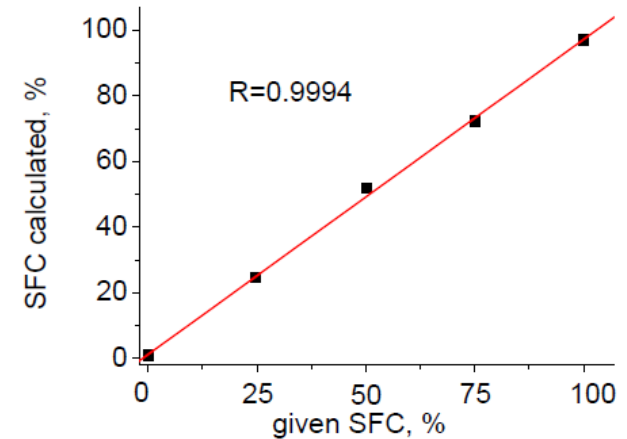
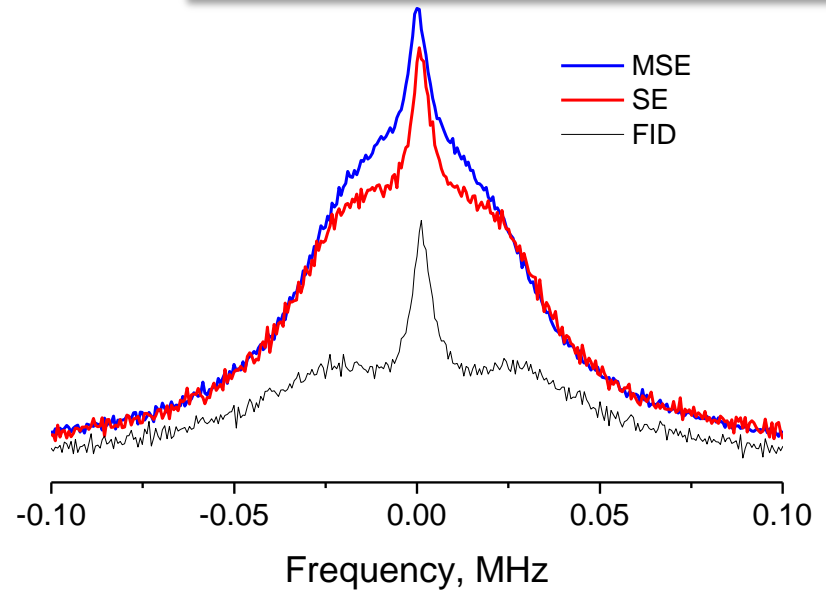
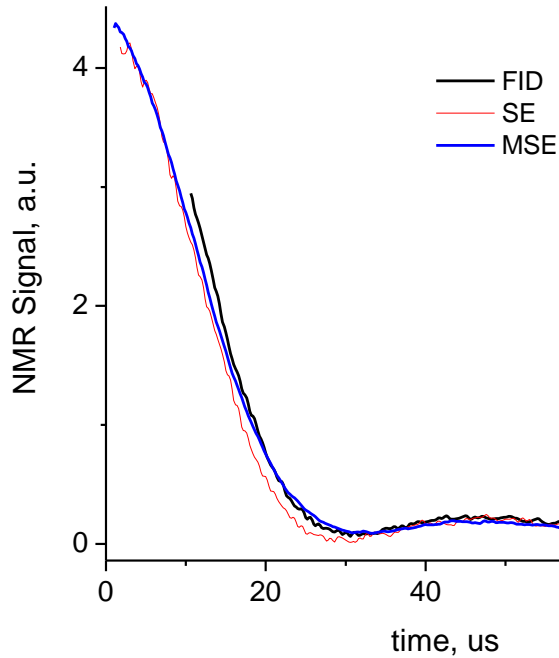
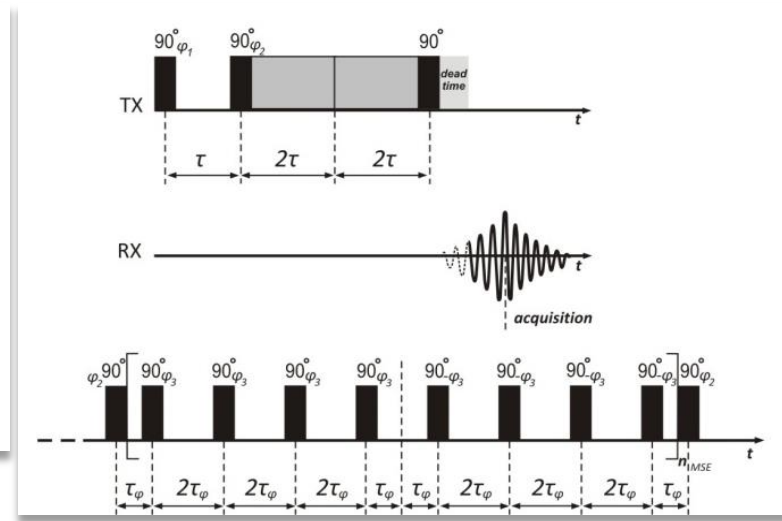
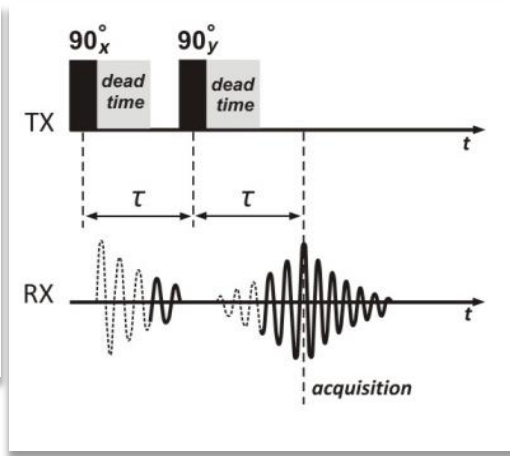
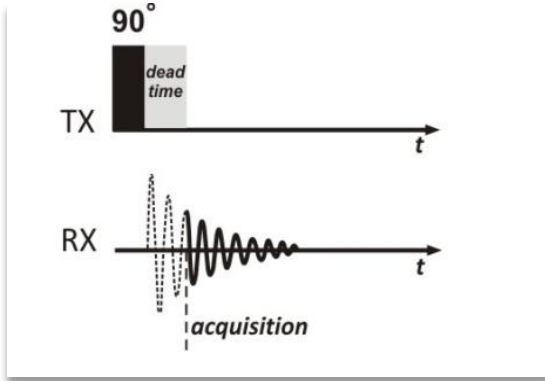


Fig. 3. Typical calibration curve of Spin Track analyzer



FID → MSE (Sihirli Sandviç Eko)

# TD-NMR: Tohumlarda Yağ ve Su Analizi

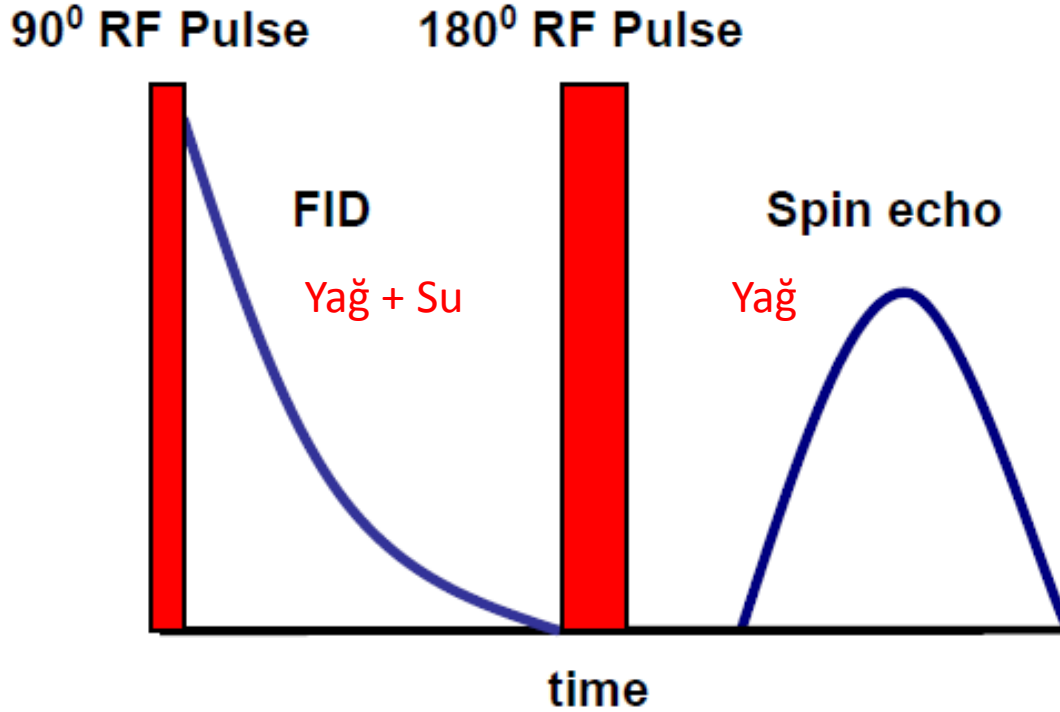
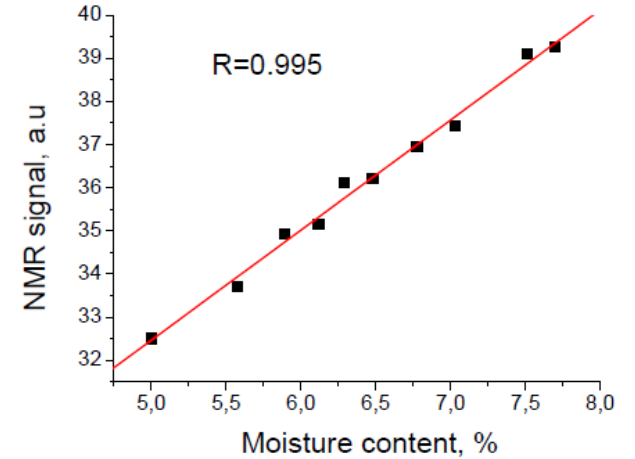
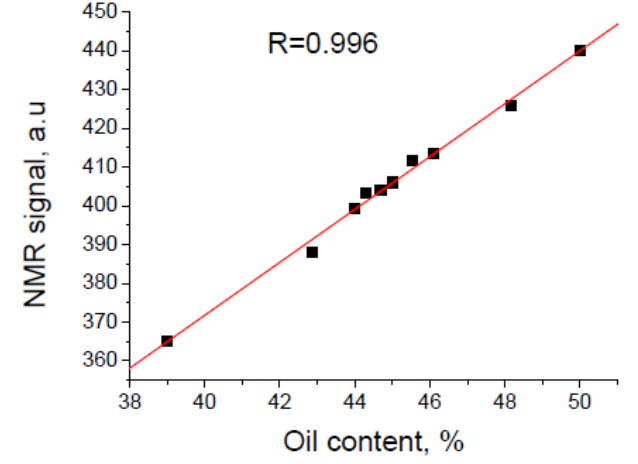


Fig 1. FID-Spin echo sequence





# Trends in Analytical Chemistry

journal homepage: [www.elsevier.com/locate/trac](http://www.elsevier.com/locate/trac)



## Compact NMR

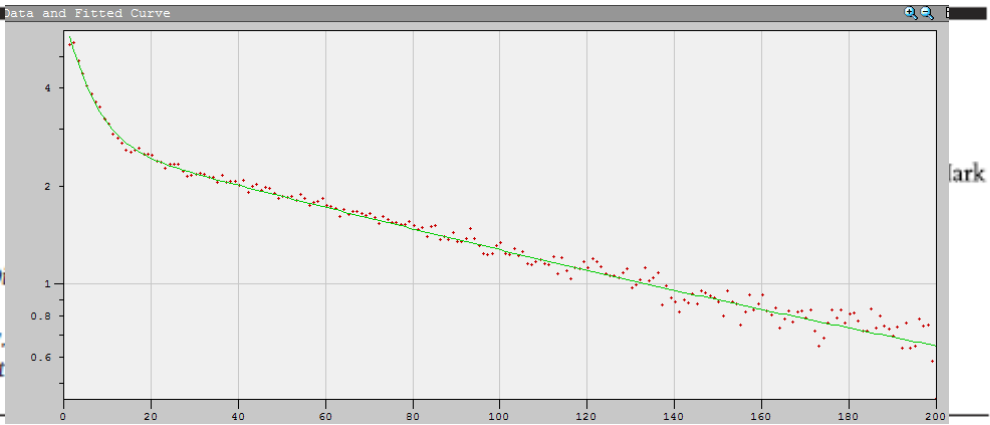
## ometry of human

Da... on <sup>a,b</sup>

<sup>a</sup> Nat... and Metabolic D...

<sup>b</sup> Dep... Molecular Biology, Greenville, Nort...

East... ratory Science... ity Institute



Files: \_CPMG14.dat

**\_CPMG14.dat**

❖ **Discrete Components Analysis**

Fitting range : [1; 200]

$\chi^2$  : 0.058

Exponential	B	B <sub>norm</sub>	$\tau$
1	3.659	0.589	2.1e+4
2	0.295	0.047	4.7e+5
3	2.260	0.364	4.7e+5

Background : 0.186

**R A C T**

magnetic reso... gy. Because it... pact instrum... on developing... specific to c... longitudinal (T<sub>1</sub>)... and lipoprote... on, insulin re... enation, altho... Current applic... and insulin re...

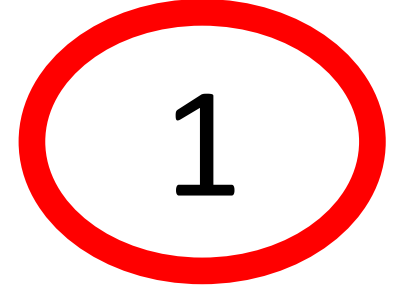
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# Kapsaisin Yüklü Nanoemülsiyonlar

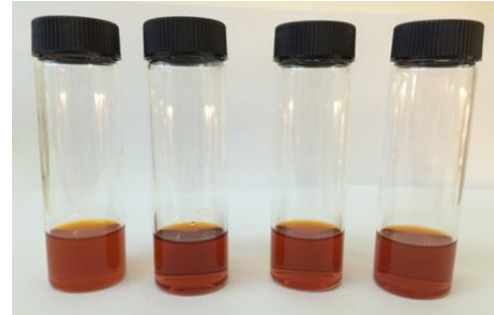


Acı biberin etken maddesi olan kapsaisin;

- ✓ Hidrofobik maddedir.
- ✓ *Birçok patojene karşı antimikrobiyal etki göstermektedir.*
  - *Staphylococcus aureus, Salmonella typhimurium, Bacillus cereus, Listeria monocytogenes, Helicobacter pylori gibi.*
- ✓ *Suda çözünürlük, termal stabilite ve biyoyararlılığını arttırmak için nanoemülsiyon kullanılmıştır.*



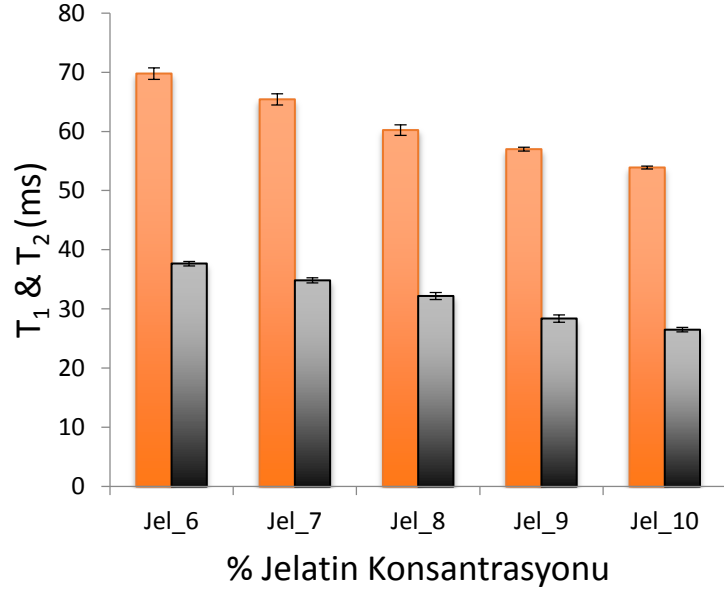
**TÜBİTAK 214O436-1002 Programı Destekli**







# Jelatin Konsantrasyonunun Yumuşak Şekerlemelerin Fiziksel Özellikleri Üzerindeki Etkisinin İncelenmesi



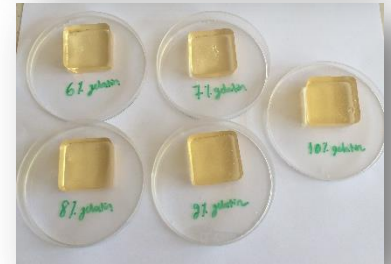
- 0.32 T NMR Sistemi

• T1

• T2

RELAKSASYON ZAMANLARI

- Yapılan boylamsal rahatlama zamanı deneylerinde %9 ve %10 benzer özellik göstermiş ve diğer numunelere göre oldukça yüksek çıkmıştır ( $p < 0.05$ ).
- Jelatin konsantrasyonu arttıkça  $T_2$  relaksasyon zamanlarında %9' dan itibaren azalış gözlemlenmiştir ( $p < 0.05$ ).



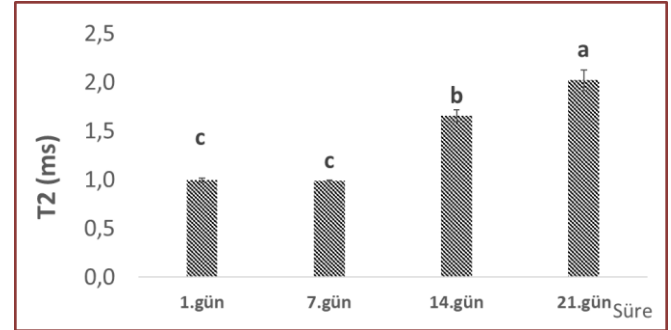
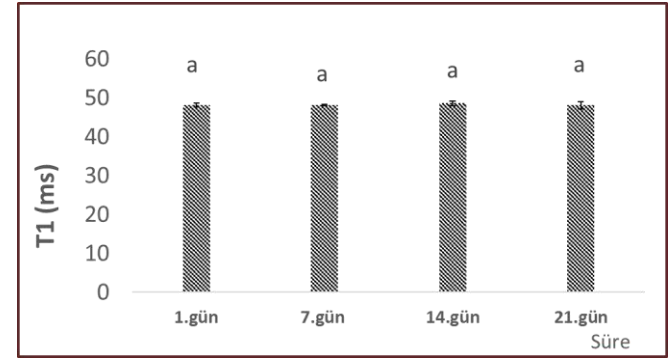
2

# Soya Proteini Ve Nişasta İçeren Yumuşak Şekerlemelerin NMR Relaksometre Kullanarak Mikroyapısal Analizi



Spin Track NMR

3



%9 nişasta ve %2 soya proteini içeren yumuşak şekerlemelerde, 14. günden itibaren T2 değerinde belirgin bir artış gözlemlenmiştir. T2 değeri numunedeki serbest su içeriği ile ilişkilendirilir, bu nedenle, zamana bağlı T2 değerindeki artış nişasta moleküllerinin sebep olduğu sineresis ile açıklanmıştır. **(TUBİTAK 1160759 2505 Programı Destekli)**

# Ayva Çekirdeđi Ekstraktının Emülsiyon Stabilitesi Üzerindeki Etki Mekanizmasının Araştırılması

4

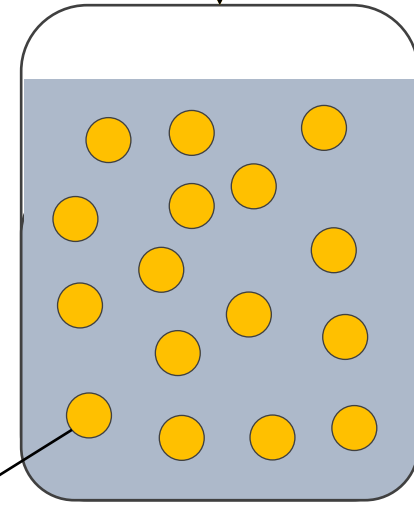
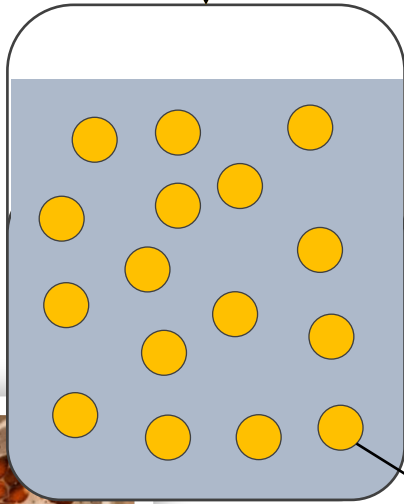
Ayva Çekirdeđi



PASP İzolesi

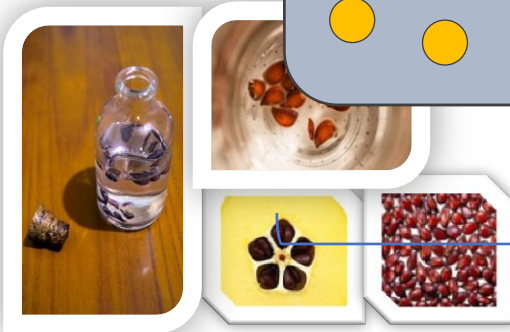


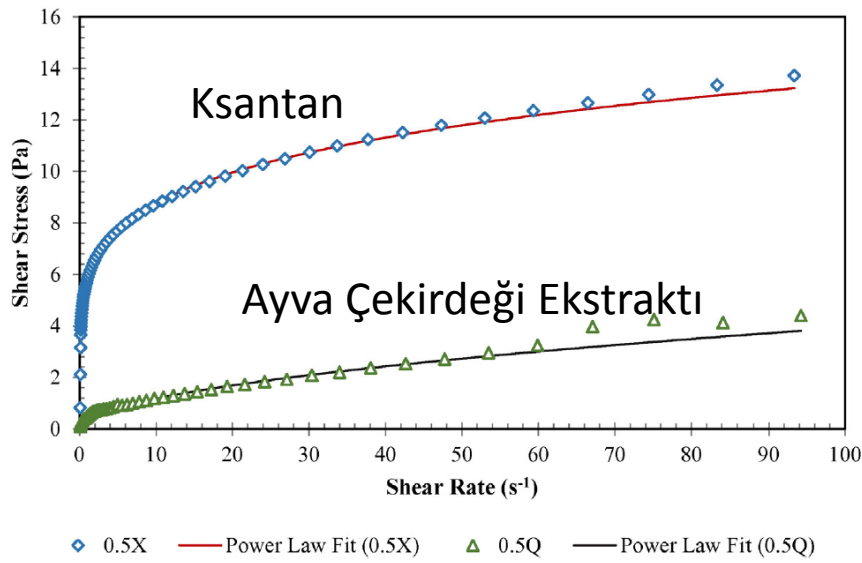
Ksantan



Ayçiçekyađı

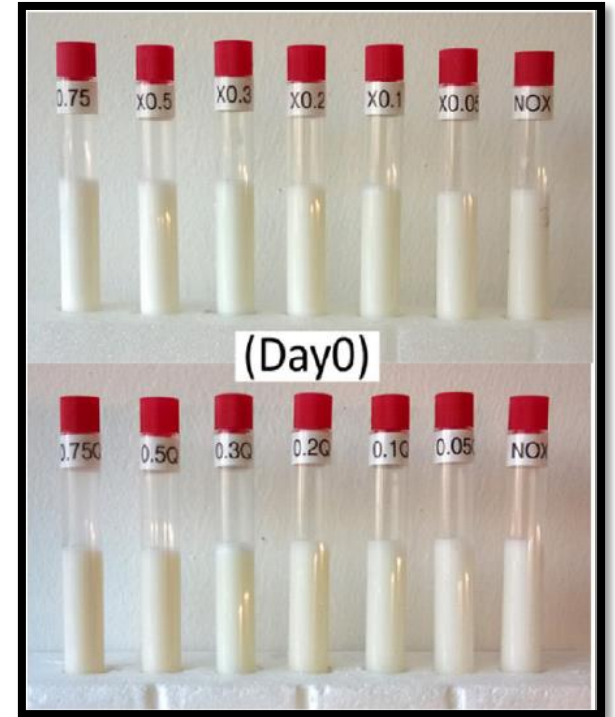
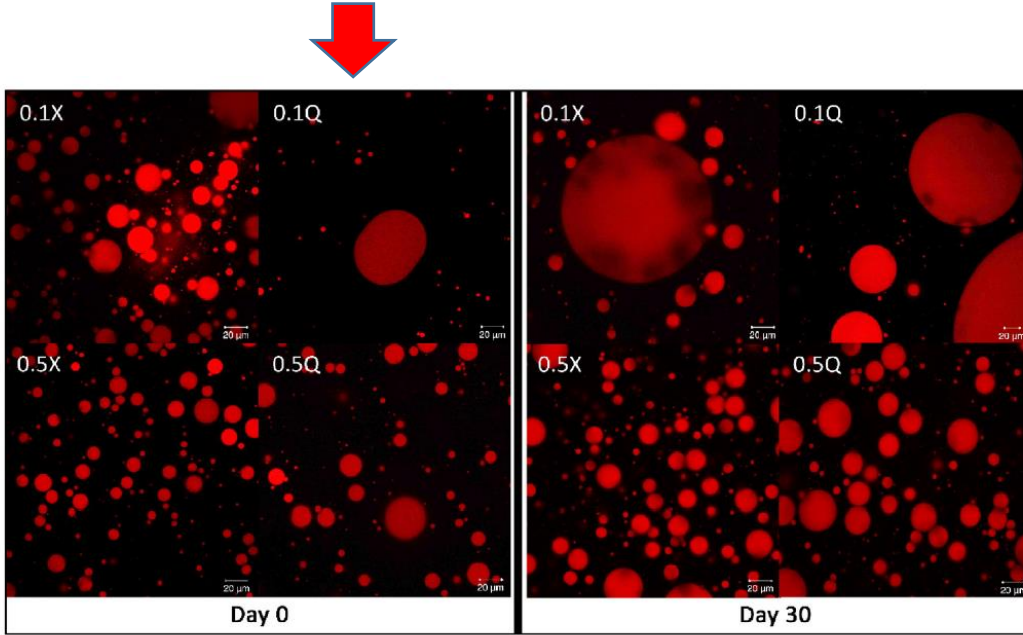
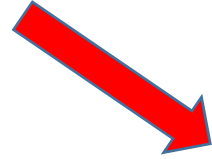
NMR Relaksometre



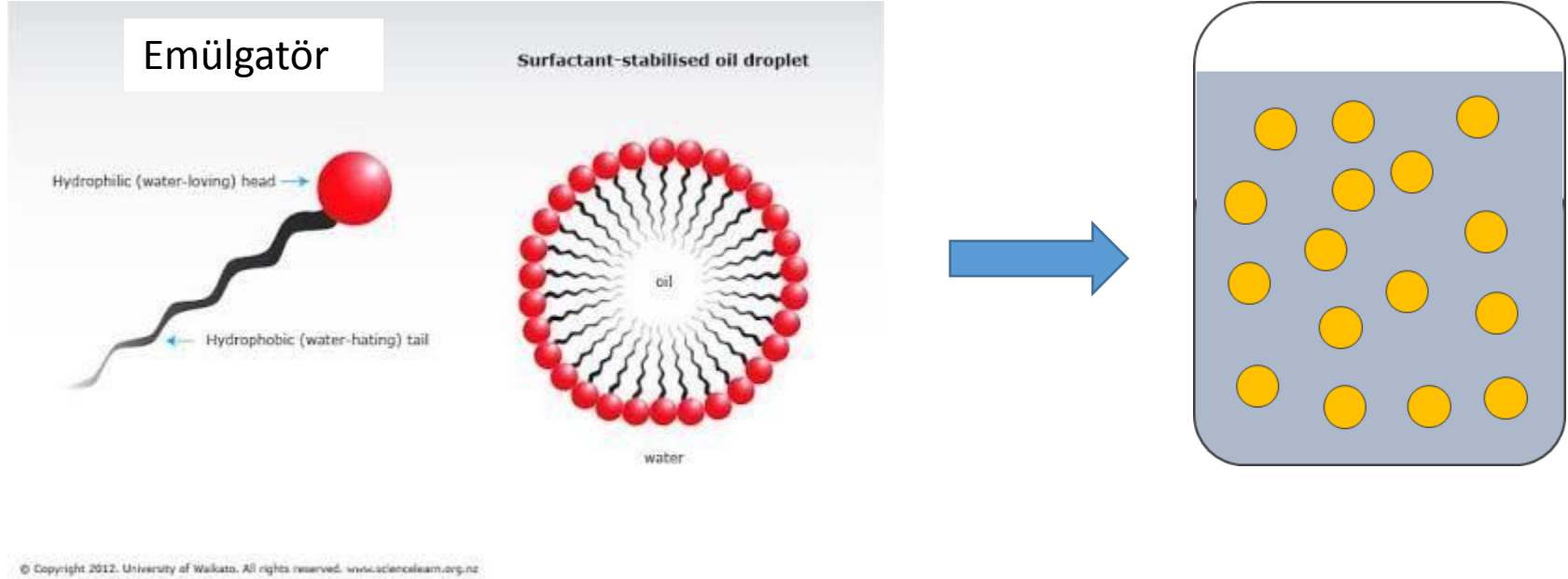


*Stabilite*  
 Ayva Çekirdeği > Ksantan

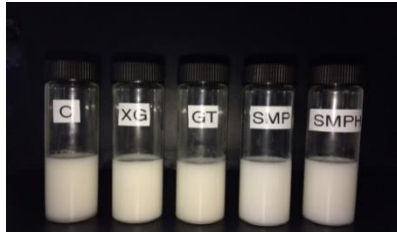
$T2_{Ksantan} > T2_{Ayva\ Çekirdeği}$



# Mekanizma



# Kitre Zamkının Emülsiyon Stabilitesi Üzerindeki Etki Mekanizmasının Araştırılması



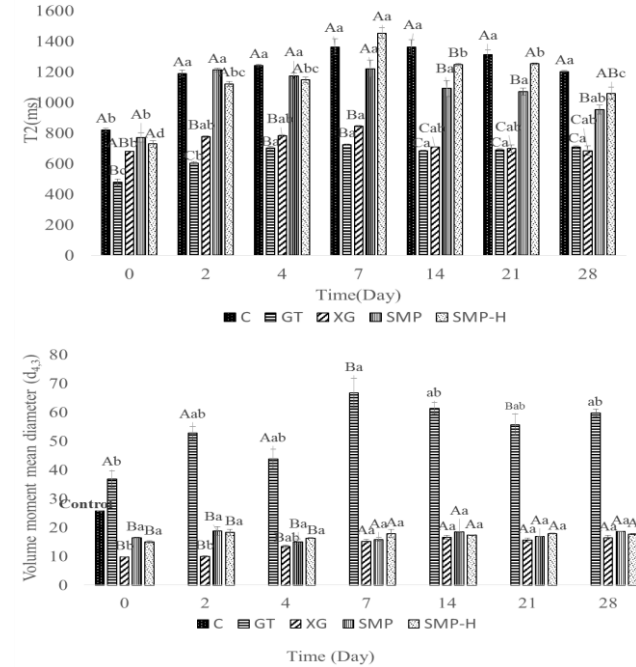
Kontrol, Ksantan zamkı, Kitre Zamkı ve SMP içeren yağ/su emülsiyonlarının görünümü



Spin Core-0.5 T NMR sistemi-T2 ölçümü



Malvern Mastersizer 3000-Parçacık Boyutu ölçümü



- En düşük T2 relaksasyon zamanı kitre zamkı ile hazırlanan emülsiyonlar için bulunmuştur ( $p < 0.05$ ) ve bu ilişki kitre zamkının yağ damlacıkları üzerindeki adsorpsiyon mekanizması ile açıklanmıştır.
- Emülsiyonların parçacık boyutu ile T2 relaksasyon zamanları arasında ters orantı olduğu gözlenmiştir.

# Yağ/Su (Y/S) Ve Su/Yağ (S/Y) Emülsiyonlarının Ayrımını NMR Relaksometre Kullanarak Tespiti

6

## Yapılan Ölçümler:

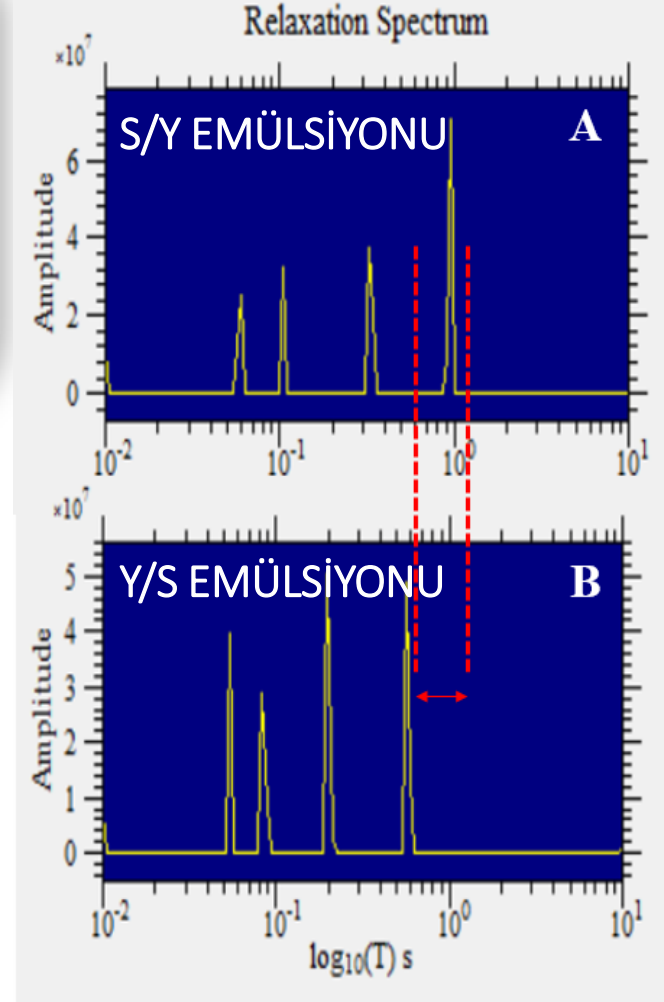
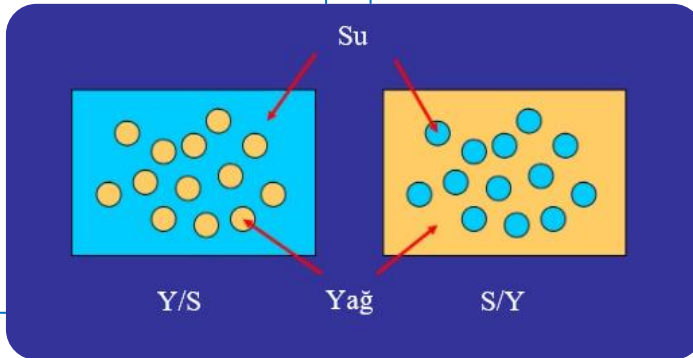


## Y/S EMÜLSİYONU

- Parçacık boyutu: 17.1  $\mu\text{m}$
- T2: 307.4 ms

## S/Y EMÜLSİYONU

- Parçacık boyutu: 0.284  $\mu\text{m}$
- T2: 611.6 ms





# Manyetik Rezonans Görüntüleme (MRG)

# Manyetik Rezonans Görüntüleme (MRG)



> 1.5 Tesla

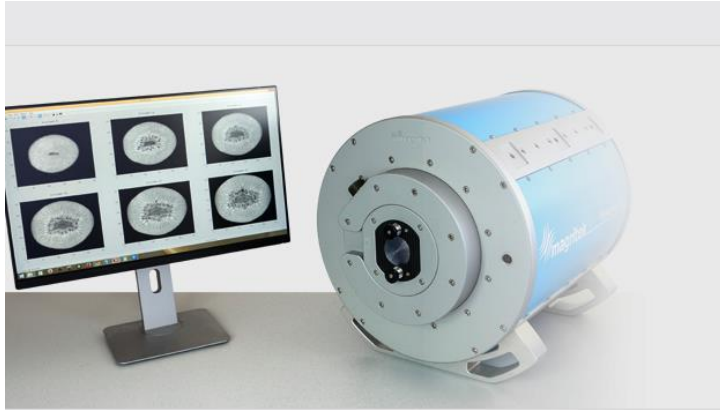


??????

# Manyetik Rezonans Görüntüleme (MRG)

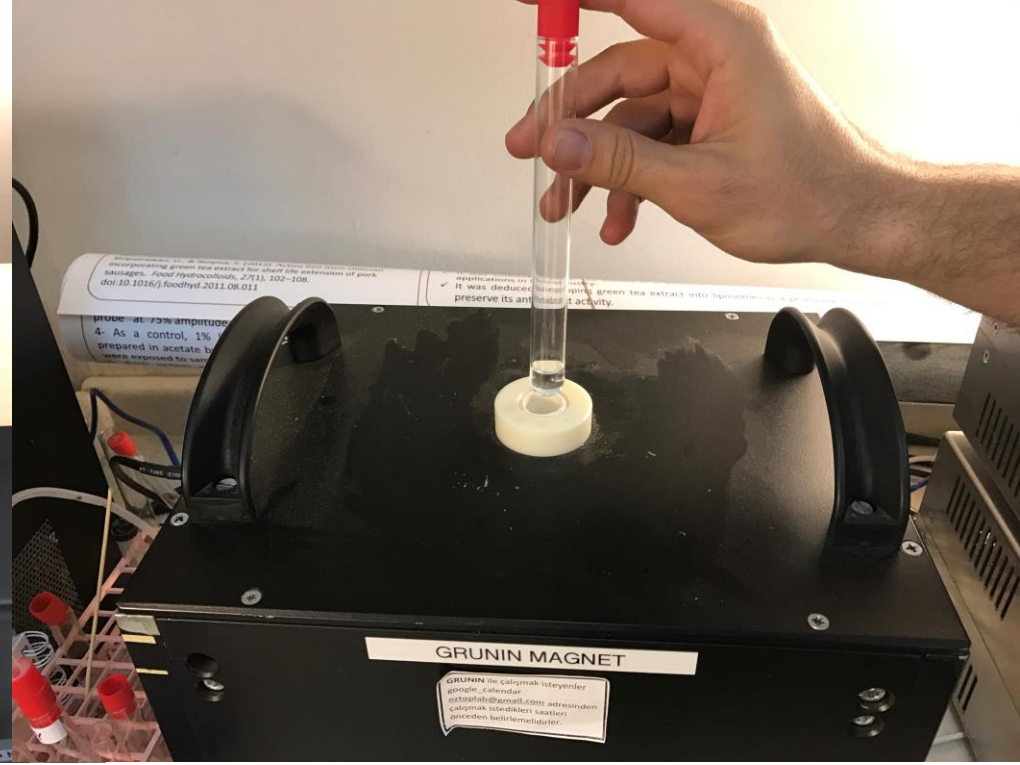
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# Düşük Manyetik Alan Gücündeki Sistemler

0.32 Tesla



# MRG veya NMR Relaksometre



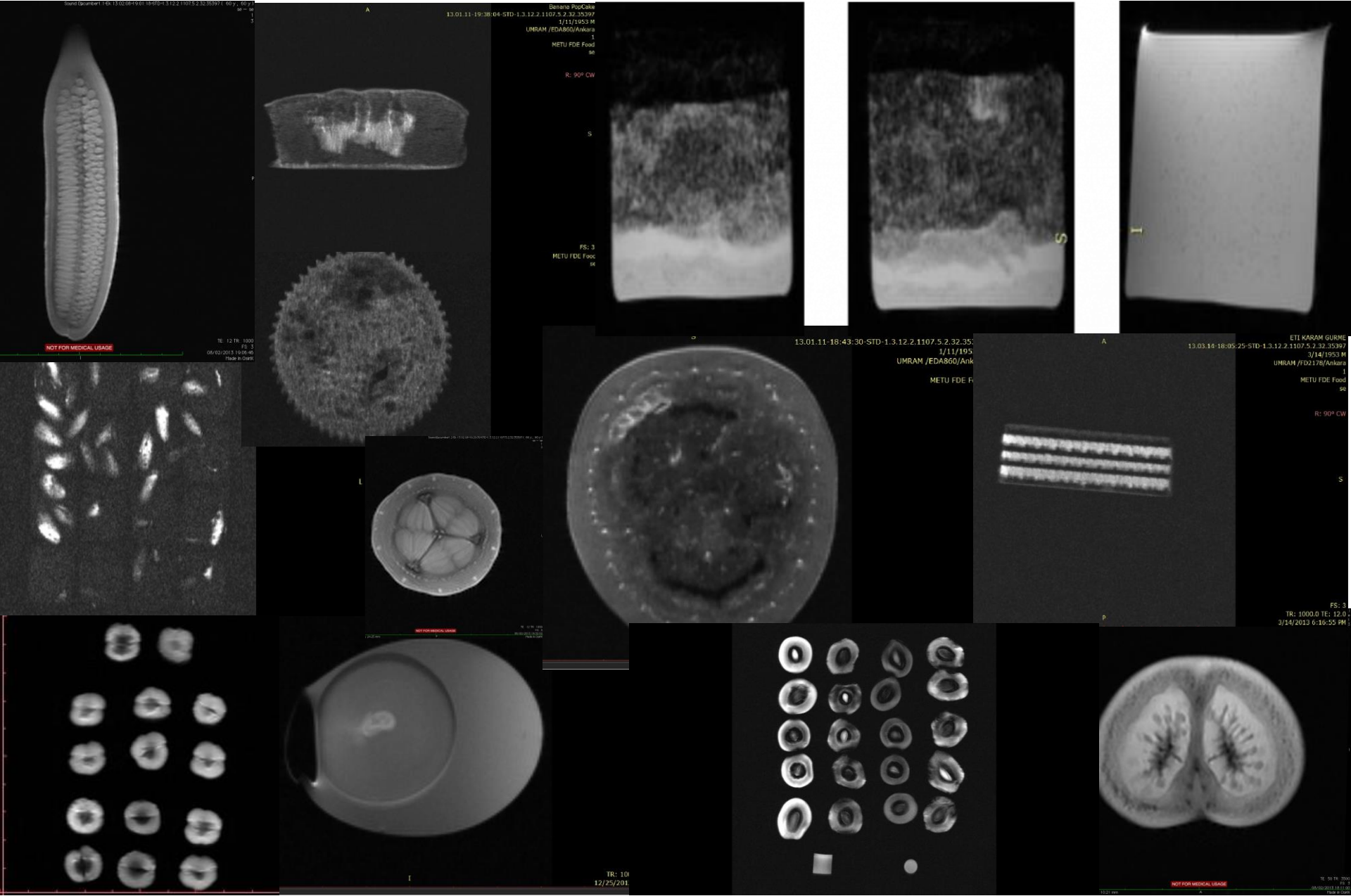
- Görüntü
- Boyutsal çözünürlük
- **Gradyentler**
- Yüksek maliyet

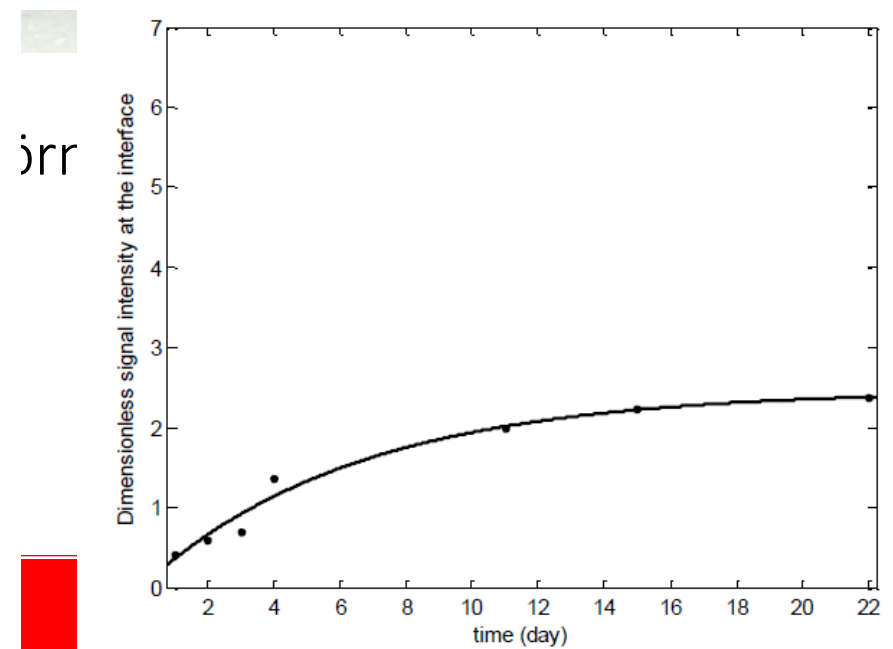
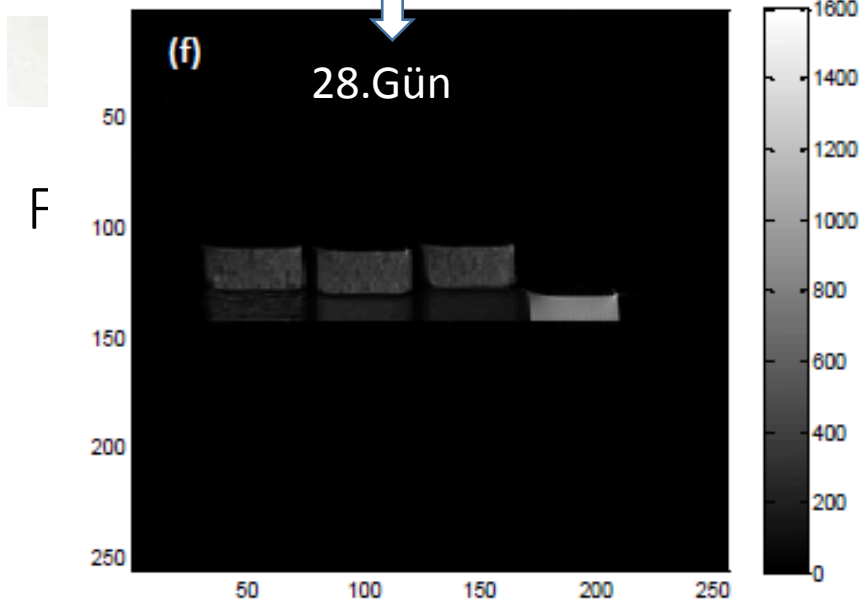
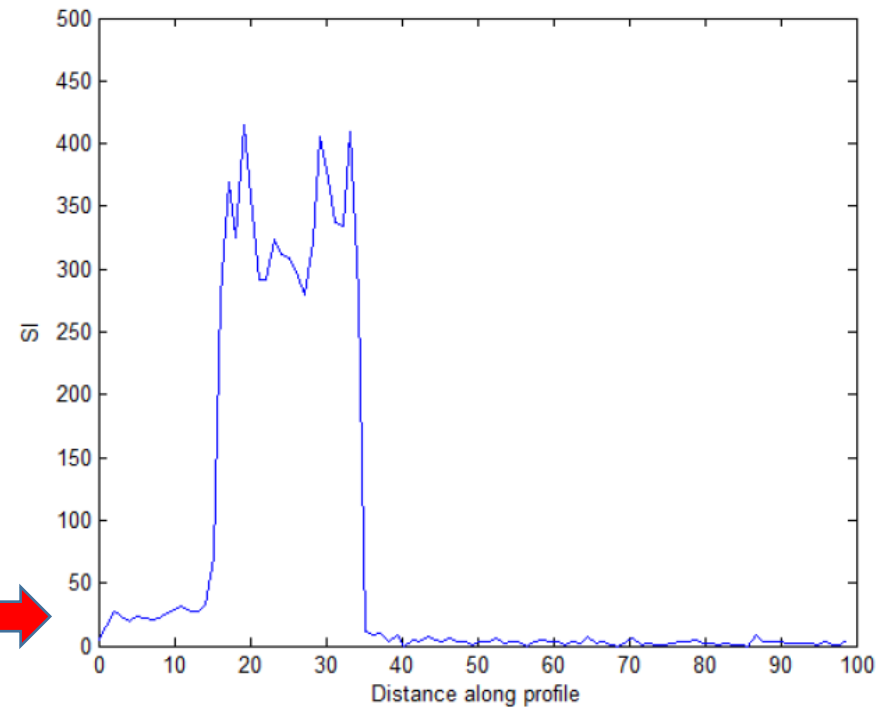
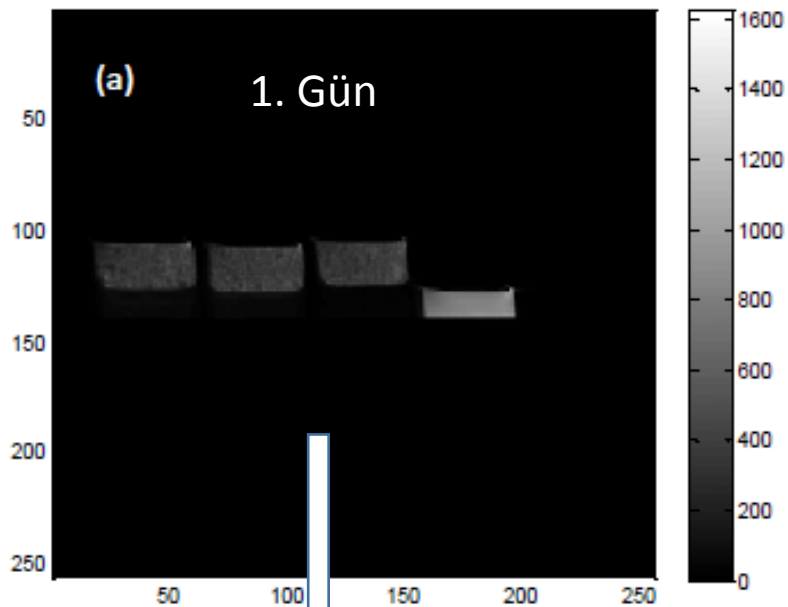
Tek yönlü sinyal  
Salınım zamanı  
Relaksometre

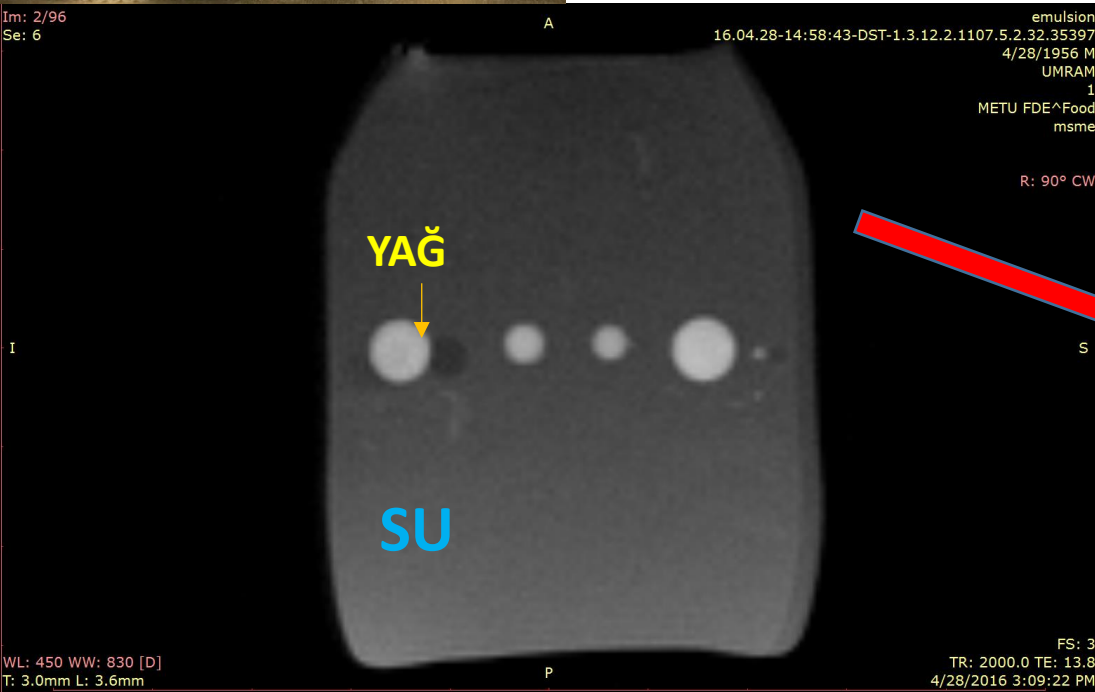
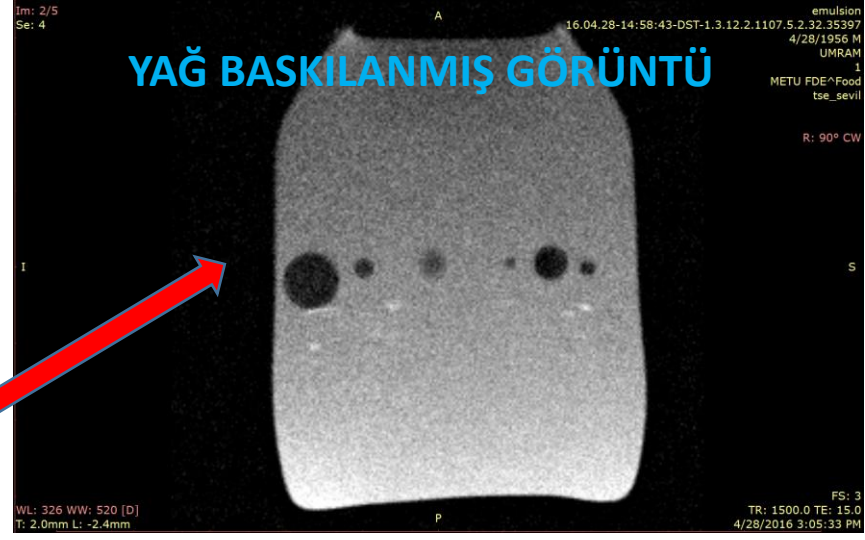
Benç tipi sistemler, düşük  
maliyet

Time Domain NMR (TD NMR)



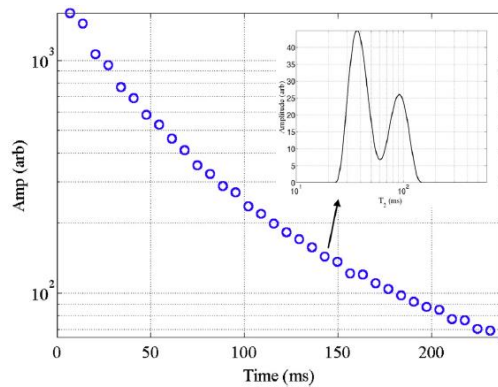
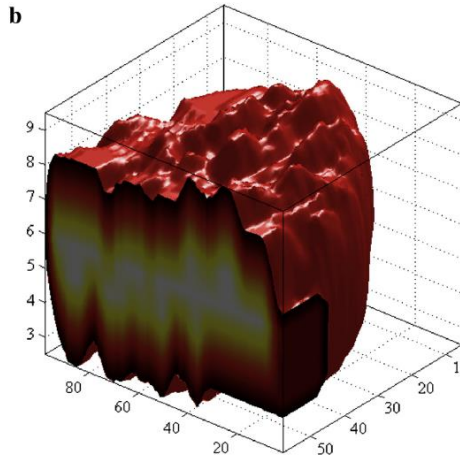
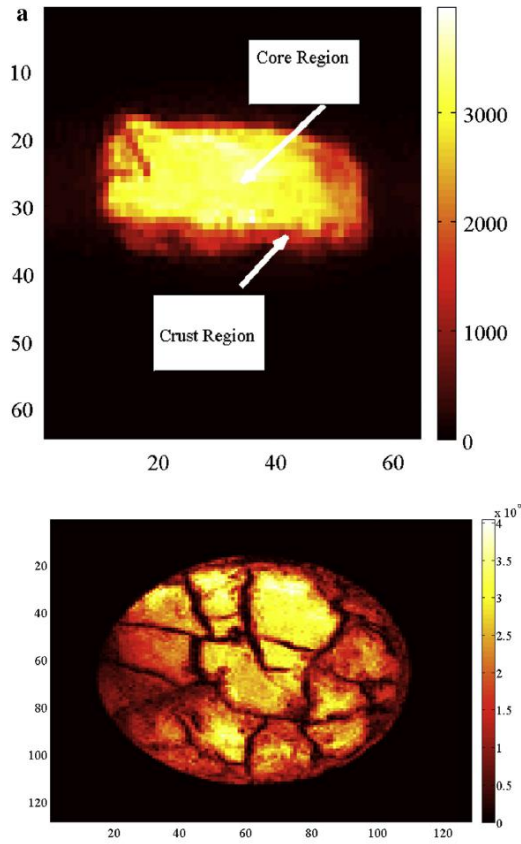








# Yağ/Su Dağılımı

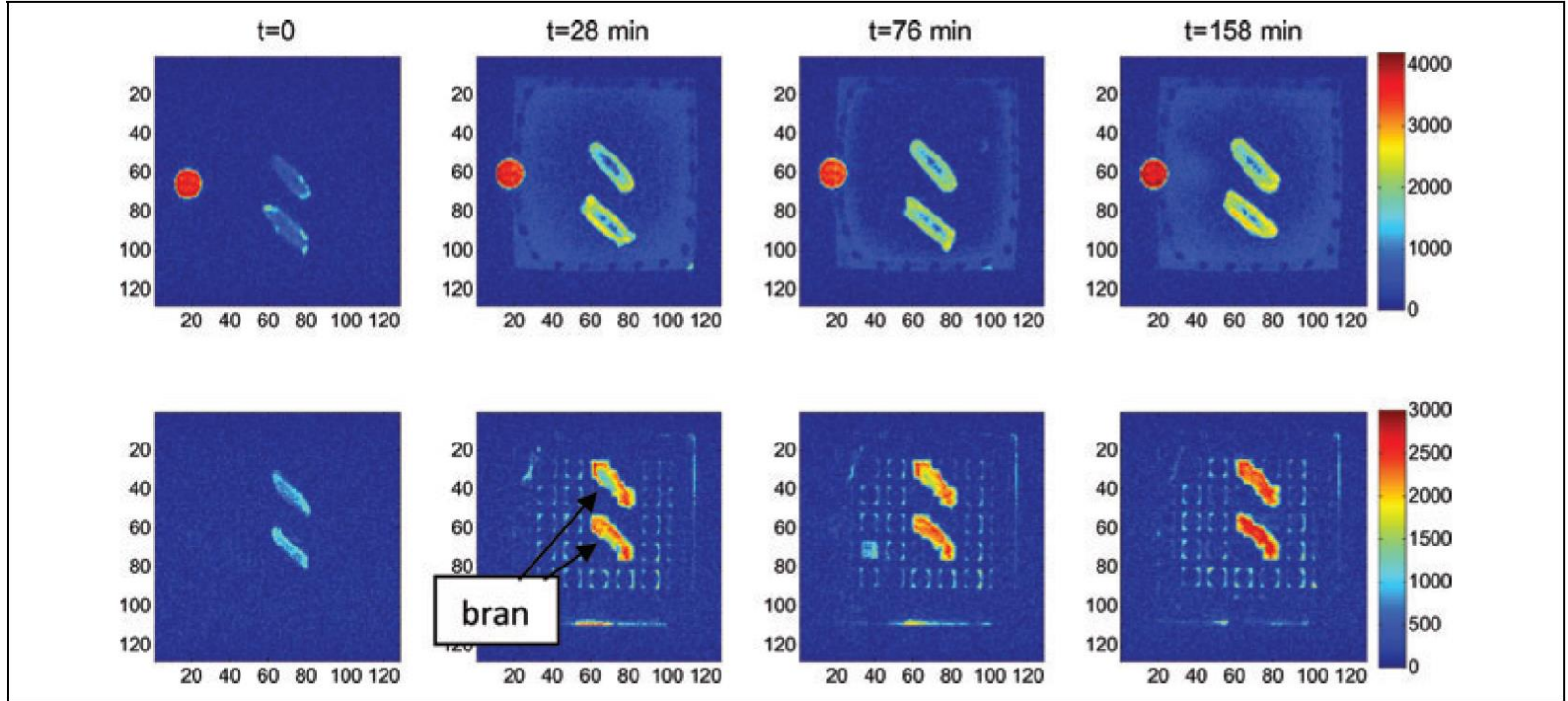


**Table 1**  
 $R^2$  (coefficient of determination) values for the relaxation spectrum parameters ( $T_{2a}$ ,  $T_{2b}$ ,  $R_{2a}$ ,  $R_{2b}$ ) and moisture and fat content of nuggets (based on linear relationships).

Coating type	Crust	Core	Coating type	Crust	Core
Methylcellulose			Control		
Temperature: 191 °C					
$T_{2a}$ vs moisture content	0.955	0.998	$T_{2a}$ vs moisture content	0.946	0.953
$T_{2a}$ vs fat content	0.675	0.979	$T_{2a}$ vs fat content	0.936	0.906
$T_{2b}$ vs moisture content	0.948	0.956	$T_{2b}$ vs moisture content	0.747	0.972
$T_{2b}$ vs fat content	0.955	0.946	$T_{2b}$ vs fat content	0.800	0.904
$R_{2a}$ vs moisture content	0.998	0.807	$R_{2a}$ vs moisture content	0.961	0.903
$R_{2a}$ vs fat content	0.836	0.728	$R_{2a}$ vs fat content	0.922	0.947
$R_{2b}$ vs moisture content	0.988	0.938	$R_{2b}$ vs moisture content	0.883	0.964
$R_{2b}$ vs fat content	0.981	0.938	$R_{2b}$ vs fat content	0.827	0.922
Temperature: 177 °C					
$T_{2a}$ vs moisture content	0.765	0.811	$T_{2a}$ vs moisture content	0.702	0.963
$T_{2a}$ vs fat content	0.995	0.920	$T_{2a}$ vs fat content	0.952	0.757
$T_{2b}$ vs moisture content	0.878	0.899	$T_{2b}$ vs moisture content	0.730	0.949
$T_{2b}$ vs fat content	0.879	0.977	$T_{2b}$ vs fat content	0.811	0.704
$R_{2a}$ vs moisture content	0.984	0.900	$R_{2a}$ vs moisture content	0.887	0.901
$R_{2a}$ vs fat content	0.675	0.979	$R_{2a}$ vs fat content	0.999	0.709
$R_{2b}$ vs moisture content	0.987	0.949	$R_{2b}$ vs moisture content	0.750	0.950
$R_{2b}$ vs fat content	0.673	0.944	$R_{2b}$ vs fat content	0.972	0.753

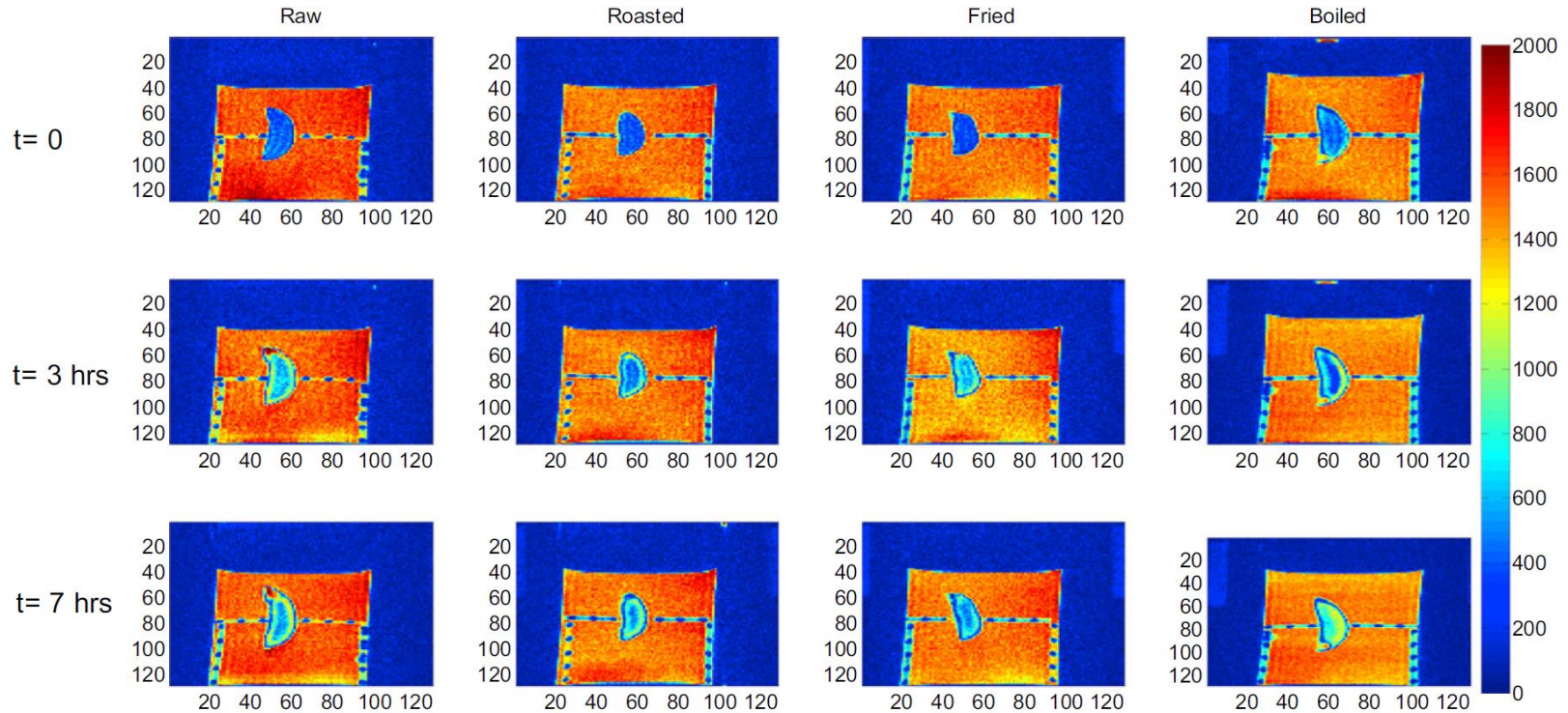
Oztop ve ark.(2014)  
 Using multi-slice-multi-echo images  
 with NMR relaxometry to assess  
 water and fat distribution in coated  
 chicken nuggets  
*LWT - Food Science and Technology*  
 55 (2014) 690-694

# MRG : Beyaz-Esmer Pirinç Sindirim



*Physical Changes in White and Brown Rice during Simulated Gastric Digestion*  
Fanbin Kong, Mecit Halil Oztop, R. Paul Singh, and Michael J. McCarthy. *Journal of Food Science* Vol. 76, Nr. 6, 2011, 450-457

# MRG : Badem Sindirim



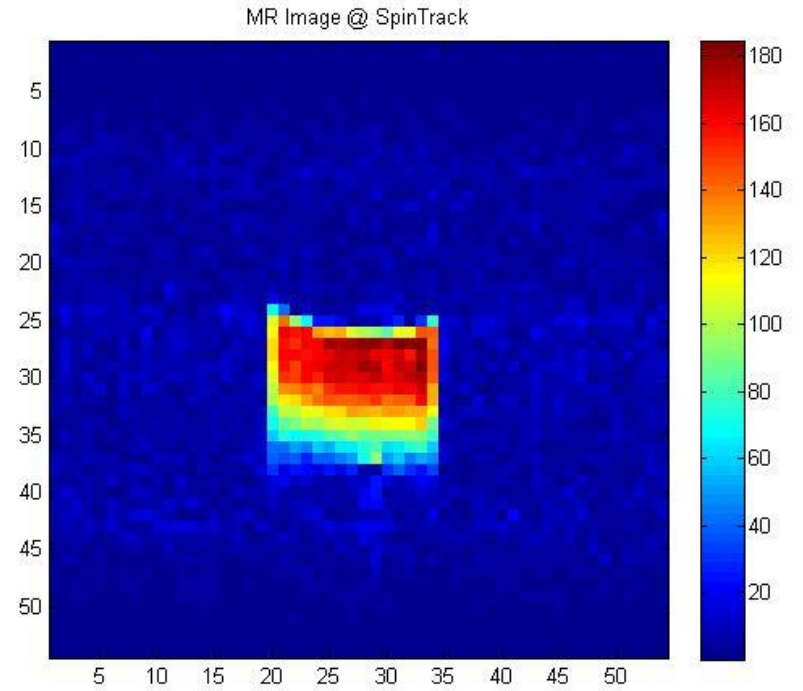
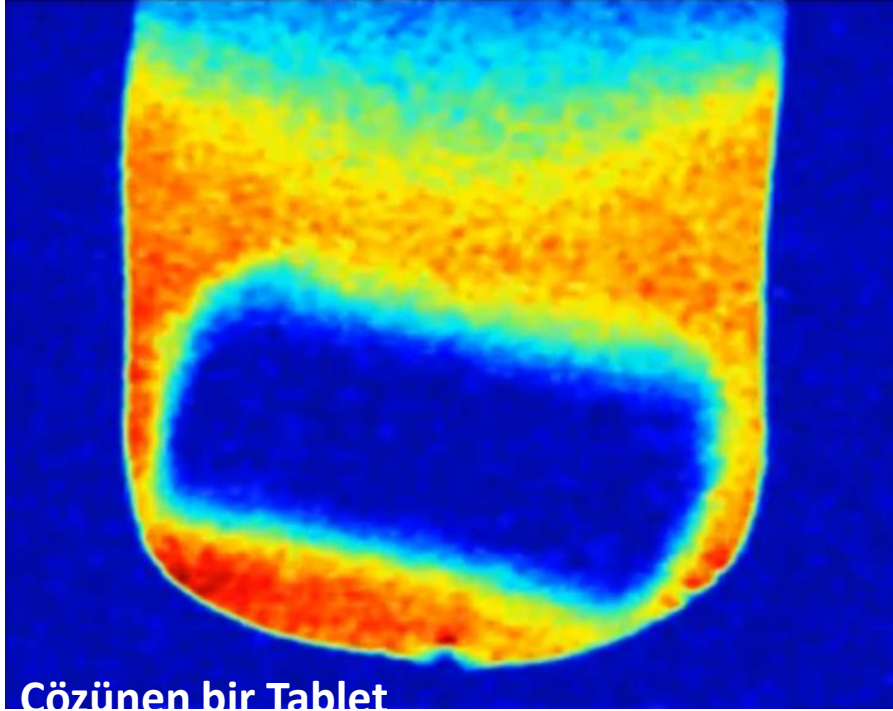
*Effect of boiling, roasting and frying on disintegration of peanuts in simulated gastric environment*

*Fanbin Kong, Mecit Halil Oztop, R. Paul Singh, Michael J. McCarthy  
LWT - Food Science and Technology 50 (2013) 32-38*

# MRG: eltiđin Kuruması



# MRG (0.5 Tesla : 10 mm)



Teşekkürler...