Texture Analyzer

application to qualify pharmaceutical products

Dr Lubna Abdalkarim Sabri

PhD Pharmaceutics, college of pharmacy, University of Baghdad

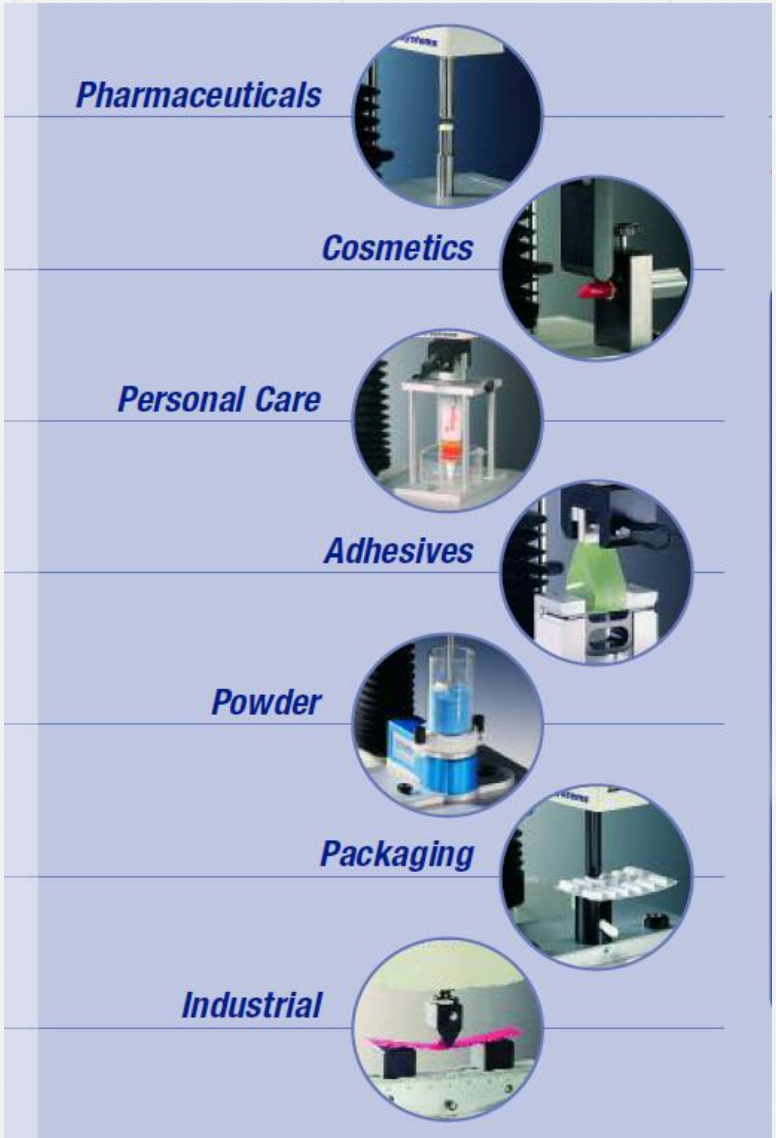
What texture can tell us?

	Toughness Tenderness Consistency Chewiness	Freshness Brittleness Stickiness Dough Quality	
Hardness Brittleness Ripeness			Stickiness Fracturability Hardness Gel Strength
	Consistency Creaminess Spreadibility		Spreadibility Adhesiveness Consistency Hardness
Resilience Break Strength Peel Tests		Tablet Strength Coating Hardness Bloom Strength Consistency	

Texture and physical properties of any compound can feel them, but---------

Can you measure them?????

- In the mid-1950, food industry, objective assessment of their products,
- Texture analysis started to use which make **Rheological characteristics quantifiable**
- General food Corporation Technical Center 1963, then develop (Insteron-texturometer)
- Texture analyzer



The TA.XT plus Texture Analyzer-
(Stable Microsystem)

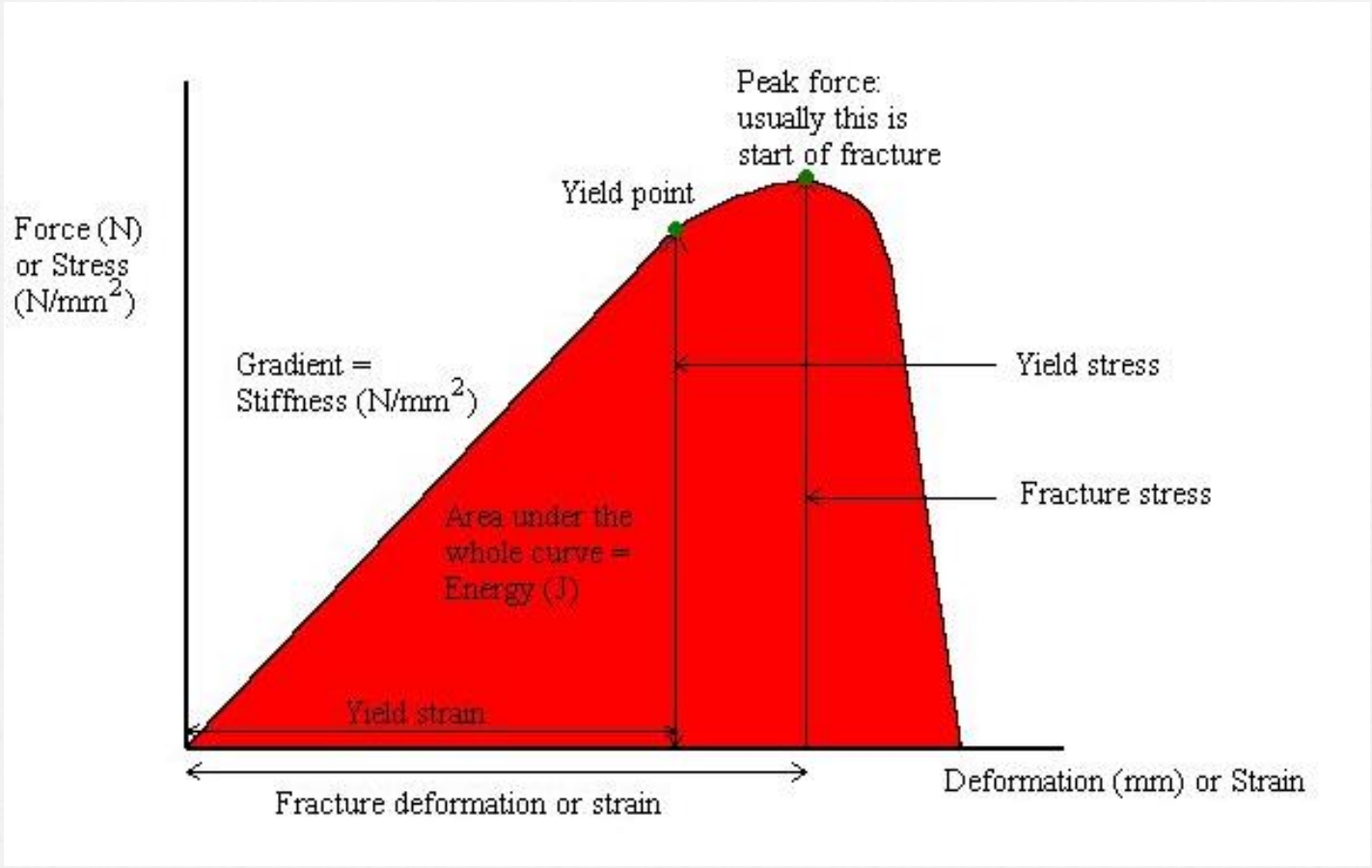
- ✓ cost-effective method to determine **the effects of** raw material or excipient quality or the **adjustment of formulation or processing** variables on end product acceptability (control the impact of changing ingredients or formulations on quality)
- ✓ Assess any problematic textural issues **occur during storage or transportation**
- ✓ effective means of **comparison with competitive products.**

A Texture Analyzer is a texture measurement system that moves up or down to compress or stretch a sample. In a simple test, the analyzer's traveling arm is fitted with a load cell.

It records the **force** / or % strain response of the sample to the **deformation** that it is undergoing by (tension, compression, bending).

To be successful, all tests depend

- ✓ the selection of the correct **testing method**,
- ✓ the manufacturing precision of **the probe** or attachment used
- ✓ the accuracy of the **analytical software** to provide the results in a clear, concise format



Basic force/deformation curve

TA.XT.plus Texture Analyser

CALIBRATION PLATFORM

LOADCELL

SAMPLE TESTING AREA

This is where the appropriate probe or fixture *(examples shown below)* is attached for sample location and testing.

Extrusion *Shearing* *Tension* *Bending* *Puncture* *Compression*

EMERGENCY STOP BUTTON

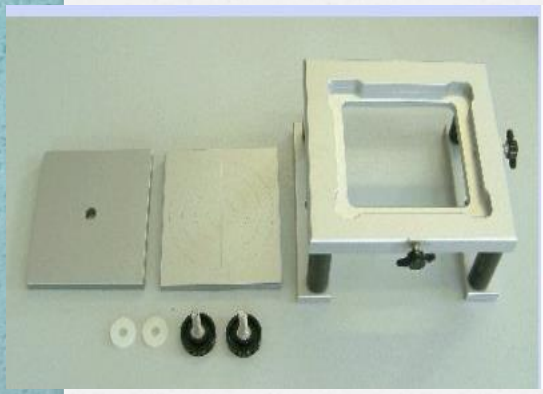
CONTROL PANEL

PC LINK

ELECTRONICS

Accessories :

HDP/90 Heavy Duty Platform



1	Heavy Duty Platform Table
1	Flat insert (target on one side)
1	Insert with 9mm fixing hole
2	Fixing screws with thumb knobs (M6x25mm)
2	Plastic washers M6

Temperature Controlled Peltier Plate (XT/PP)

The Peltier controlled Peltier Plate provides a stable surface temperature for testing products small or thin products such as pressure sensitive adhesives. This ensures that temperature effects are either minimised or are able to be accurately investigated.
The surface dimensions are 110mm x 100mm and maximum operating temperature is +80C to a minimum of 30C below ambient.

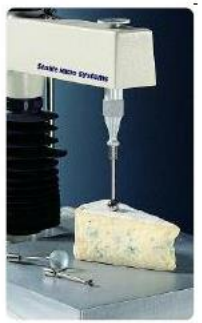
Temperature Controlled Peltier Cabinet (XT/PC)

The **Peltier Cabinet** provides a temperature controlled environment using PID control with an operating range from +80°C to 20°C below ambient. It is fixed directly to the base of the texture analyser via nylon insulating pillars that provide a thermal barrier from the instrument. Samples can be allowed to equilibrate to the required temperature before testing is performed within the cabinet. A double walled transparent hinged door allows the sample to be seen during testing and is easily opened for sample access. A Peltier Control Unit is provided on which temperature is set and displayed.

A total internal height of 85mm provides a testing area suitable for many typical sample sizes but may be limited if testing a large sample or if the test requires attachment of a large probe or fixture.

Probes

Cylinder Probes



Spherical Probes

Blister Pack Support
A/BP

Metered Dose Inhaler Support Rig A/IS

Conical Probes

TTC Spreadability Rig
HDP/SR

Granule Compaction Rig*
HDP/GCR

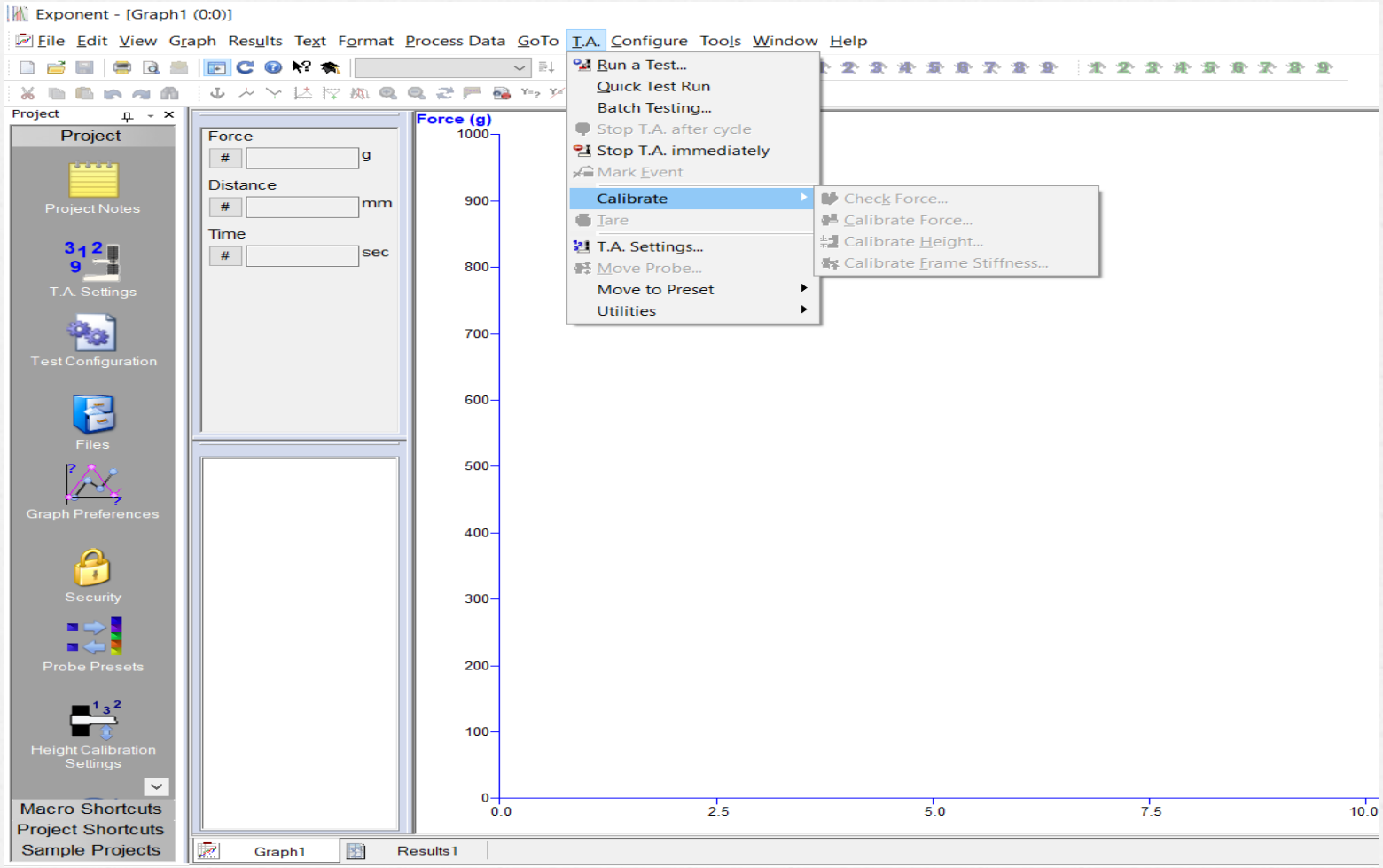
Powder Compaction Rig* - Low Tolerance
A/PCR & A/PCRS

Tablet Disintegration Rig*
A/TDR

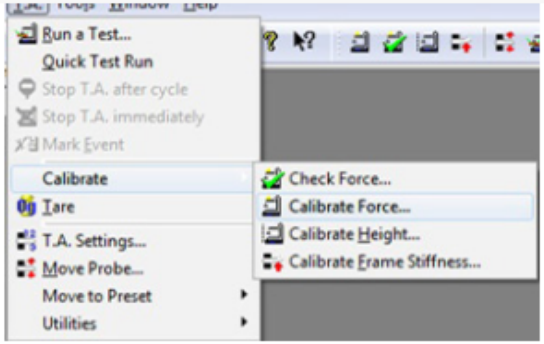
Multiple Puncture Probe
A/MPP

Needle Probe
P/2N

Calibration and T.A setting

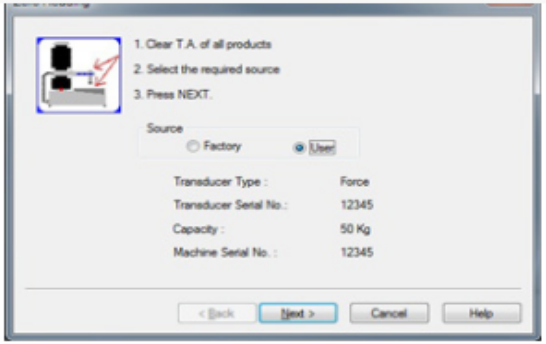


* Standard Operating Procedures (SOPs): Texture Analyzer TA-Xtplus
https://www.youtube.com/watch?v=WUc5yFqAkVA



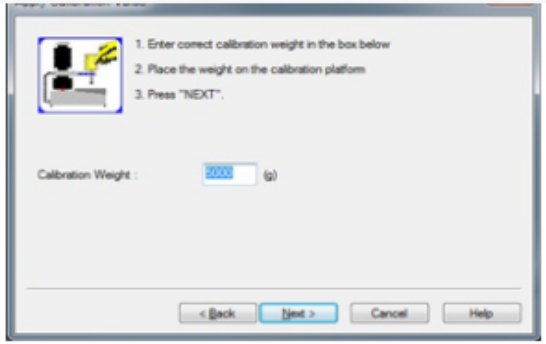
1

From the tool bar menu, click on *T.A.* > *Calibrate* > *Calibrate Force*.



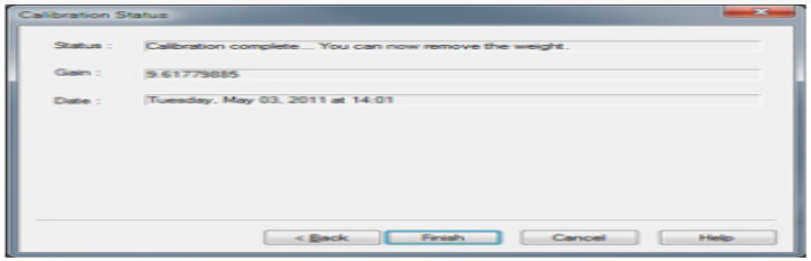
2

Select either *Factory* or *User*, as required. The installed loadcell capacity is identified.



3

Select the calibration weight of your choice.

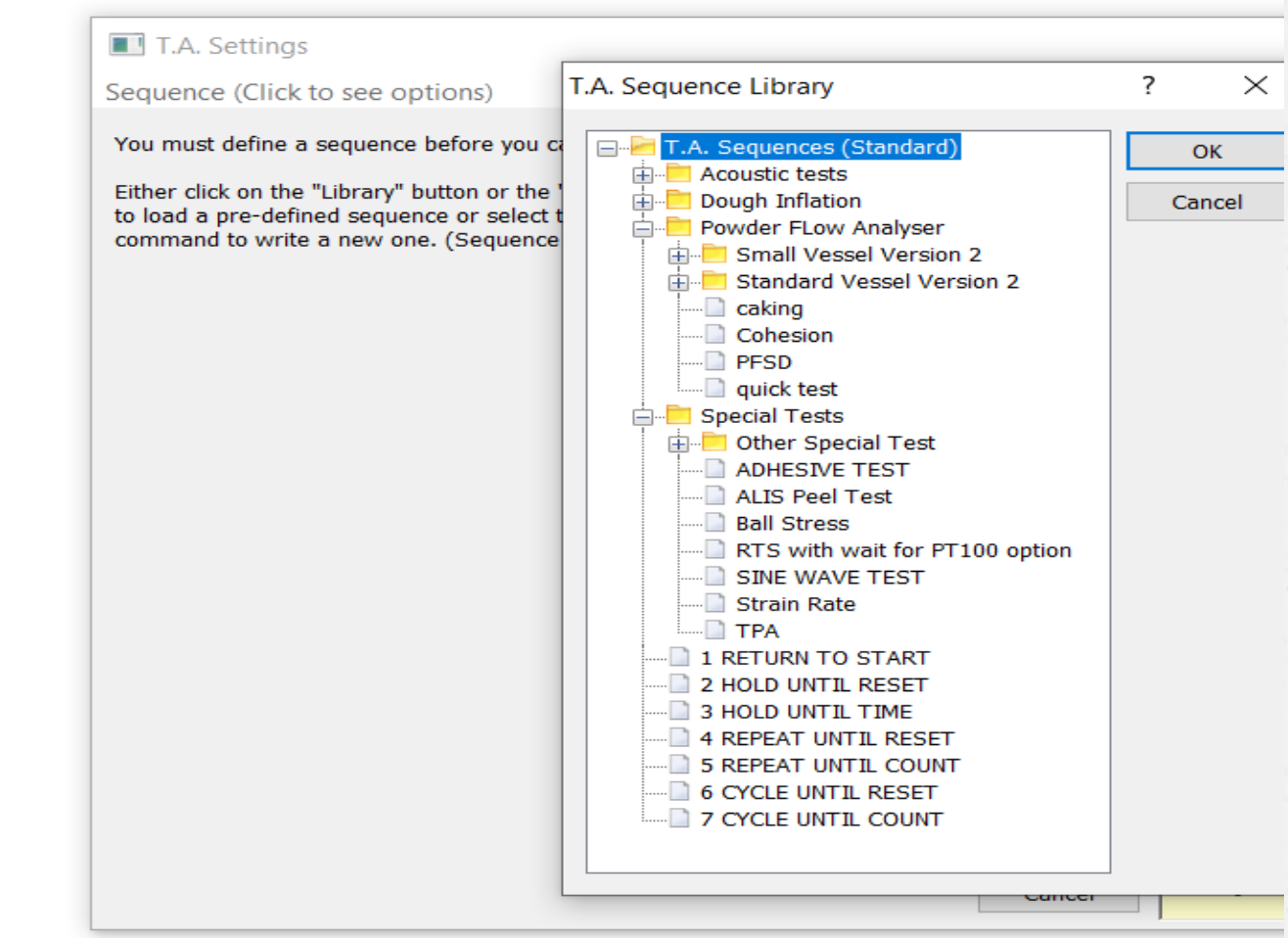


4

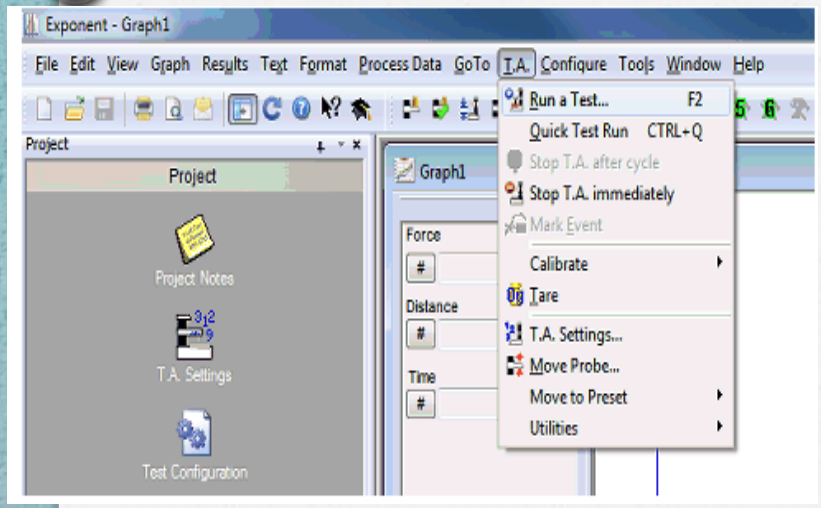
This window should be displayed. Remove the weight from the calibration platform.

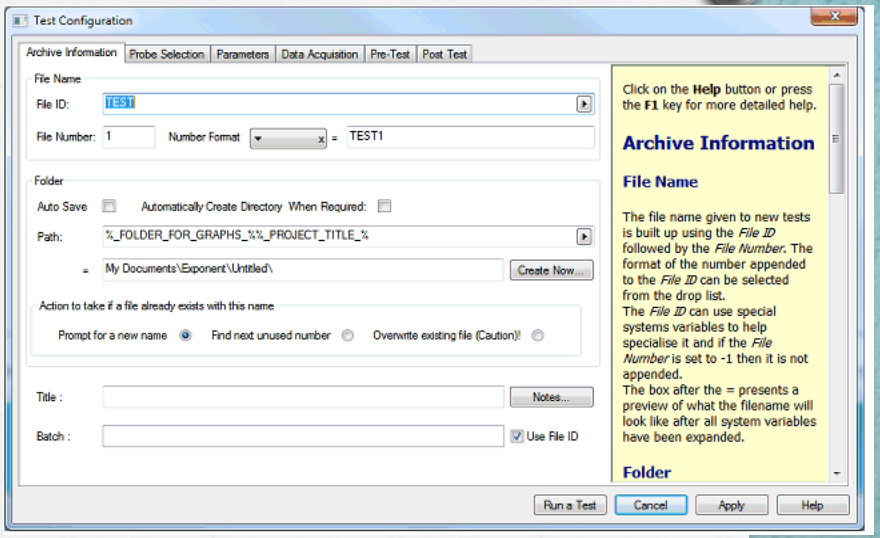
T.A Setting

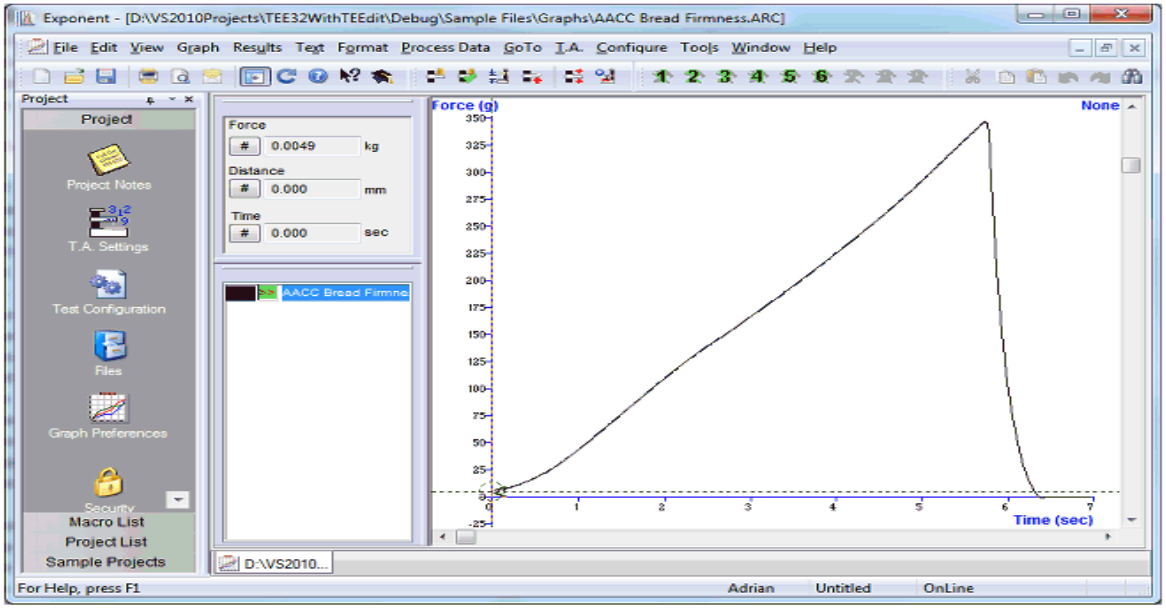
From Library A series of classical TA tests are are available for selection

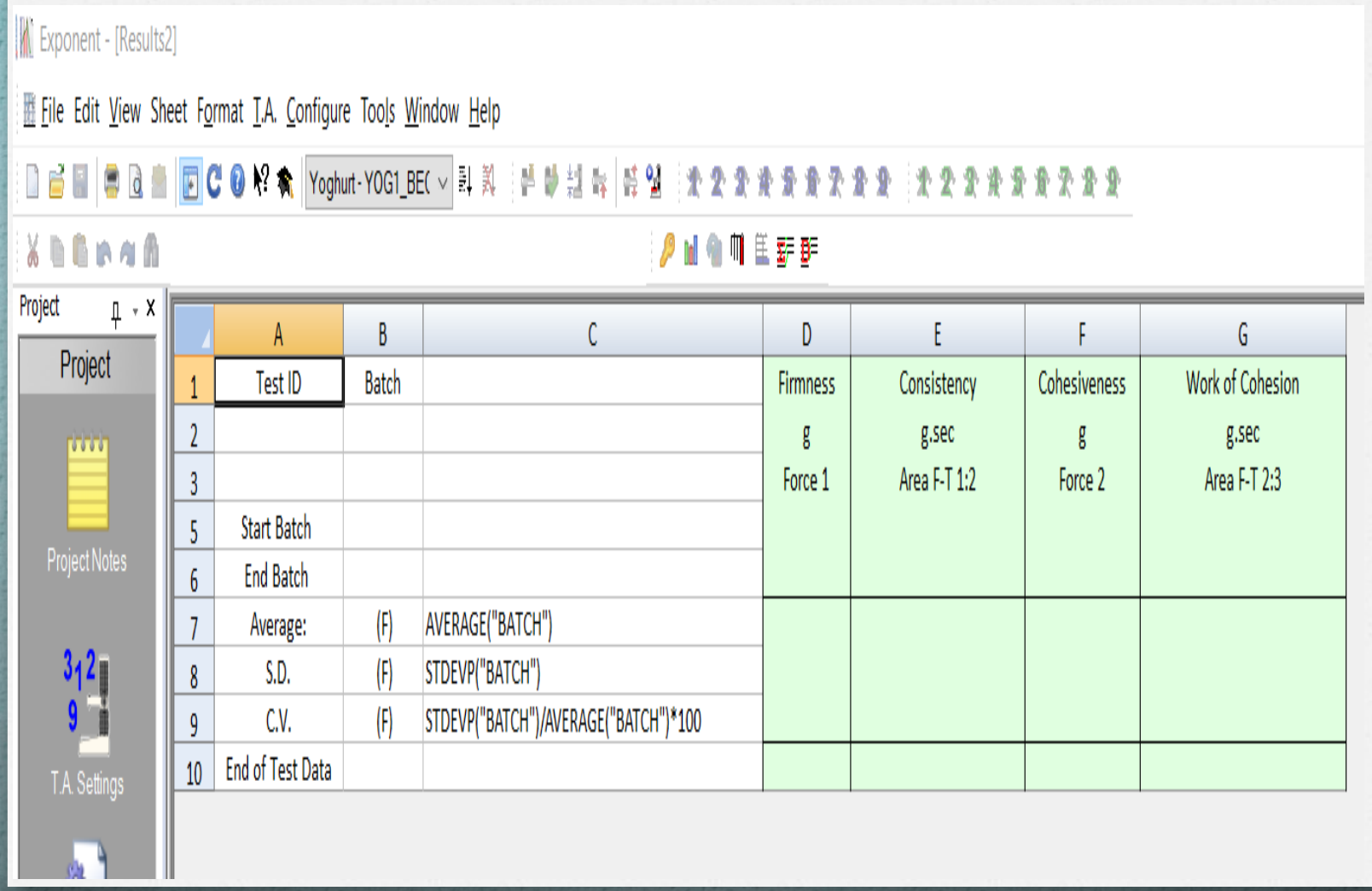


Running Test









	A	B	C	D	E	F	G
1	Test ID	Batch		Firmness	Consistency	Cohesiveness	Work of Cohesion
2				g	g.sec	g	g.sec
3				Force 1	Area F-T 1:2	Force 2	Area F-T 2:3
5	Start Batch						
6	End Batch						
7	Average:	(F)	AVERAGE("BATCH")				
8	S.D.	(F)	STDEVP("BATCH")				
9	C.V.	(F)	STDEVP("BATCH")/AVERAGE("BATCH")*100				
10	End of Test Data						

Textural properties of Topical Formulations

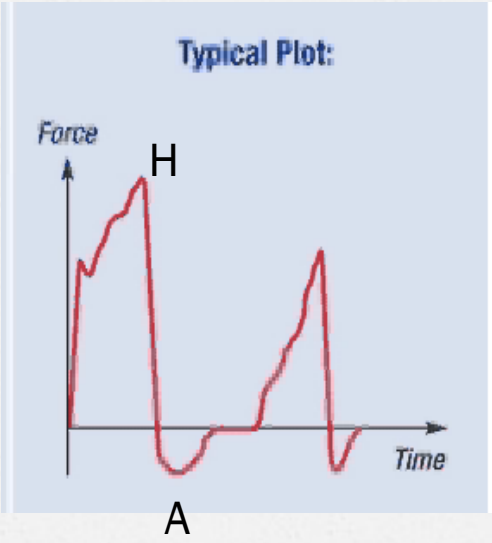
"Evaluation of in vitro mucoadhesiveness and texture profile analysis of doxycycline in situ hydrogels"

- The mechanical properties or texture properties obtained from operating TPA2 mode and analyzed for hardness, compressibility, cohesiveness and adhesiveness

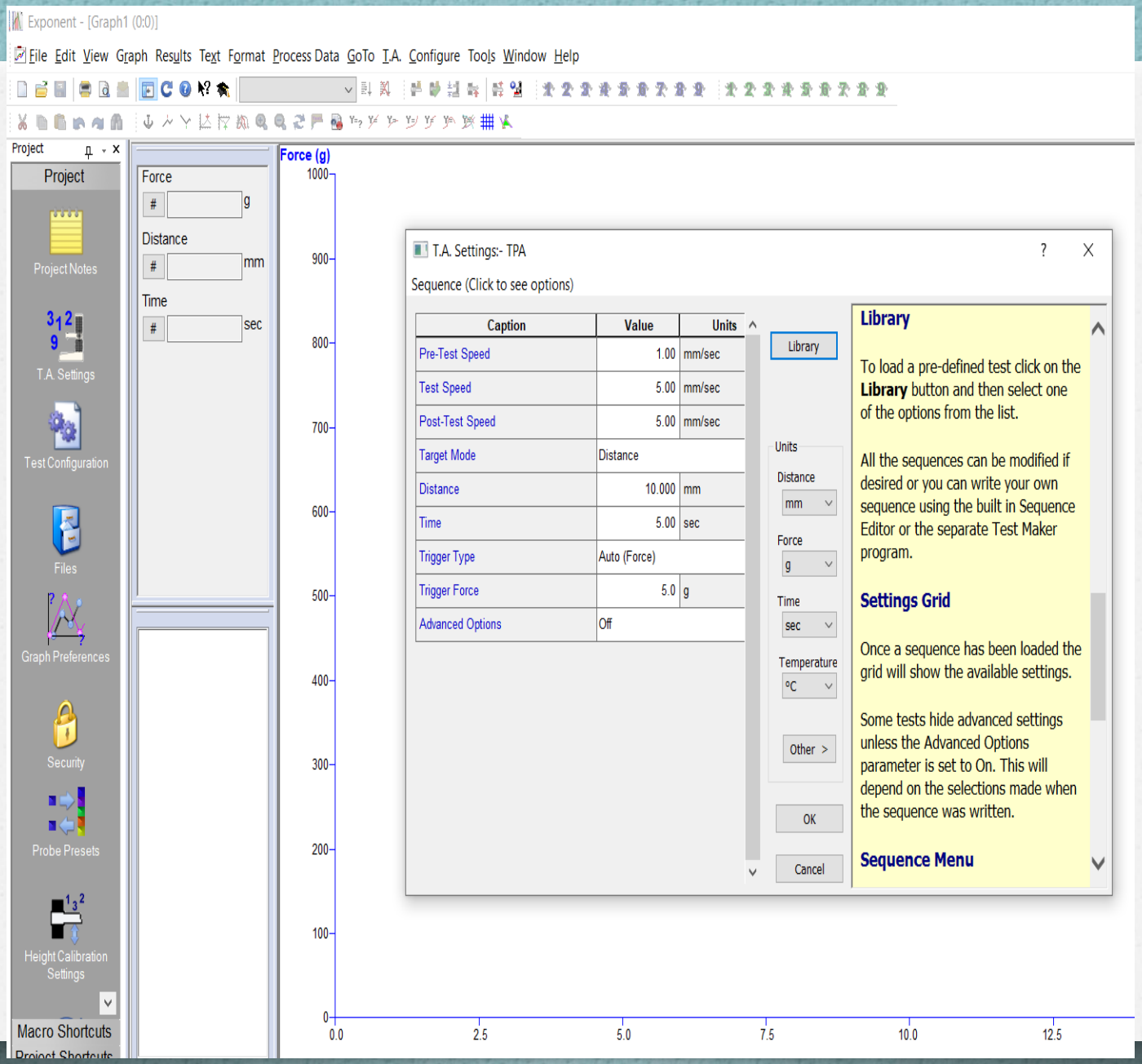
Library ------- specific test------TPA

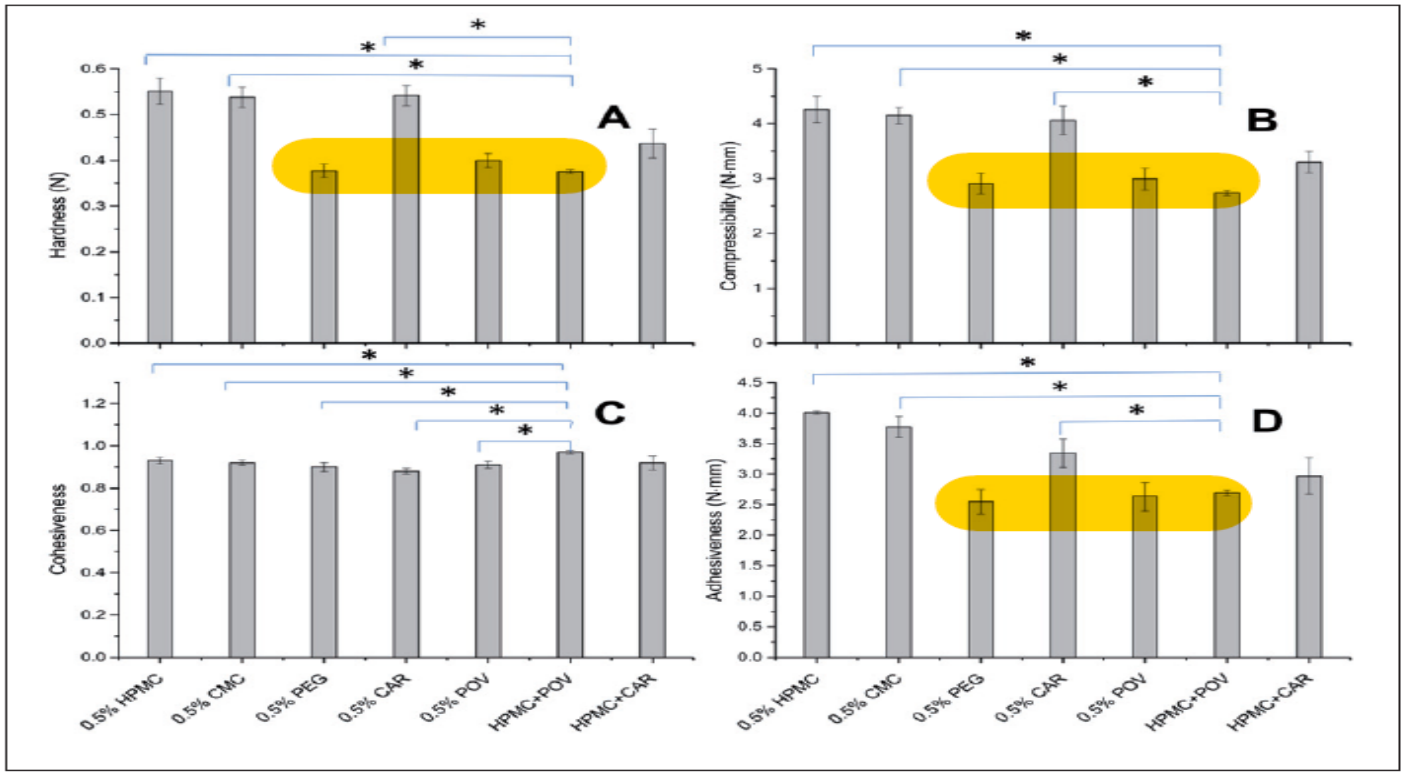
P/10, P/35 cylindrical probe (perspex , graphite, stain steel) twice compress sample

- ✓ Hardness
- ✓ Adhesiveness
- ✓ Compressibility
- ✓ cohesiveness



*V. G. R. PATLOLLA et al. Pharmazie 75 (2020)


Exponent - [Graph1 (0:0)]
File Edit View Graph Results Text Format Process Data GoTo T.A. Configure Tools Window Help
Project
Project
Project Notes
T.A. Settings
Test Configuration
Files
Graph Preferences
Security
Probe Presets
Height Calibration Settings
Macro Shortcuts
Force
g
Distance
mm
Time
sec
Force (g)
1000
900
800
700
600
500
400
300
200
100
0
0.0
2.5
5.0
7.5
10.0
12.5
T.A. Settings:- TPA
Sequence (Click to see options)
Caption
Value
Units
Pre-Test Speed
1.00
mm/sec
Test Speed
5.00
mm/sec
Post-Test Speed
5.00
mm/sec
Target Mode
Distance
Distance
10.000
mm
Time
5.00
sec
Trigger Type
Auto (Force)
Trigger Force
5.0
g
Advanced Options
Off
Library
Units
Distance
mm
Force
g
Time
sec
Temperature
°C
Other >
OK
Cancel
Library
To load a pre-defined test click on the Library button and then select one of the options from the list.
All the sequences can be modified if desired or you can write your own sequence using the built in Sequence Editor or the separate Test Maker program.
Settings Grid
Once a sequence has been loaded the grid will show the available settings.
Some tests hide advanced settings unless the Advanced Options parameter is set to On. This will depend on the selections made when the sequence was written.
Sequence Menu



*$p<0.05$, One way ANOVA Turkey HSD *post hoc* test

Fig. 3: Texture profile analysis where the comparison of hardness(A), compressibility (B), cohesiveness (C)and adhesiveness (D) are measured for hydrogels containing different mucoadhesive polymers: HPMC = hydroxypropyl methyl cellulose; CMC = carboxymethyl cellulose, PEG = polyethylene glycol 6000, CAR = Carbopol 974P, POV = Povidone, HPMC+POV = 0.25% HPMC and 0.25% Povidone, HPMC+CAR = 0.25% HPMC and 0.25% Carbopol 974P

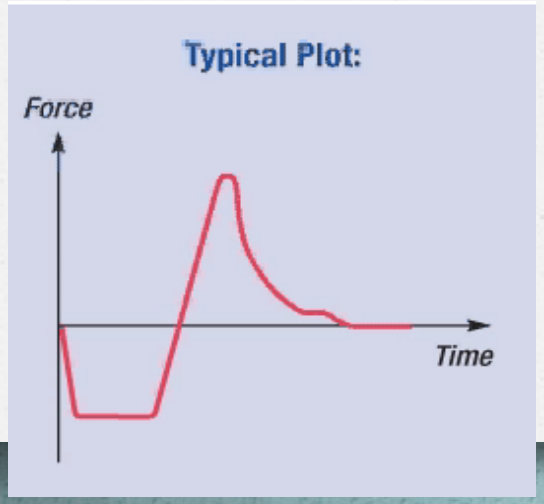
In vitro mucoadhesion

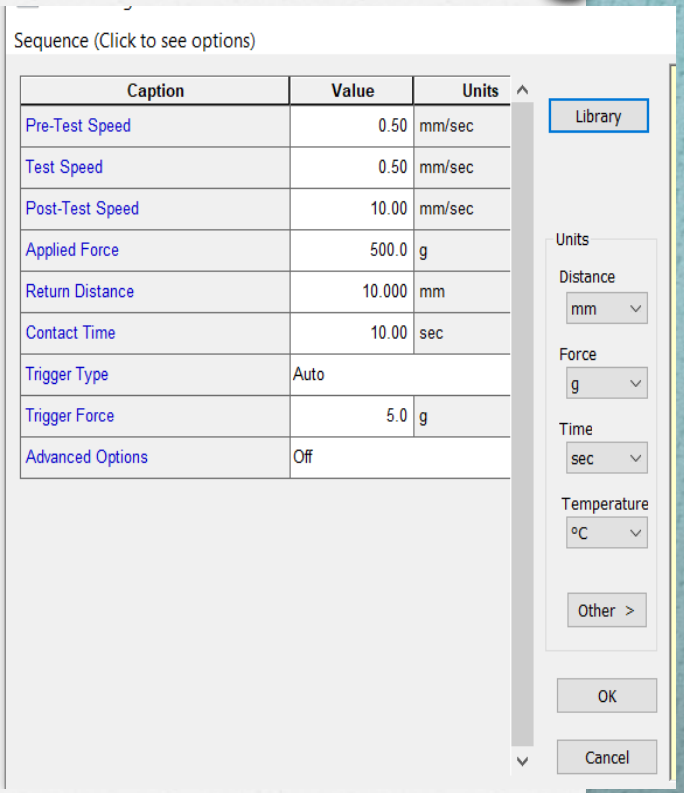
The force required to detach the artificial mucus membrane from the surface of hydrogel

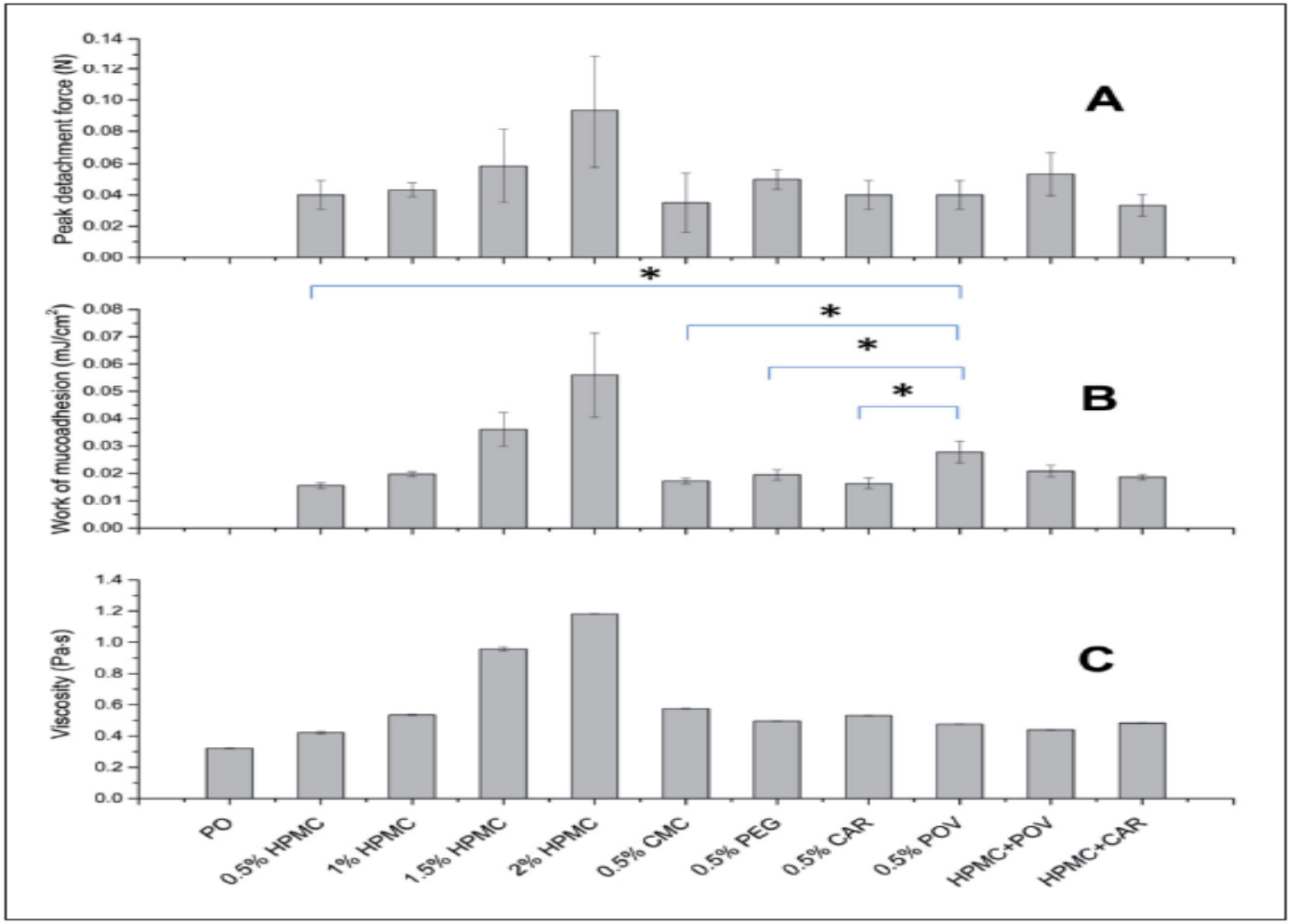
The AUC values obtained from force-time curve were converted to force-distance.

$$\text{Work of mucoadhesion } \frac{AUC}{\pi r^2} = \frac{N \cdot mm}{cm^2} = \left(\frac{mJ}{cm^2}\right)$$

Tensile strength was measured as the peak detachment force required to detach the test hydrogel from the mucosa.





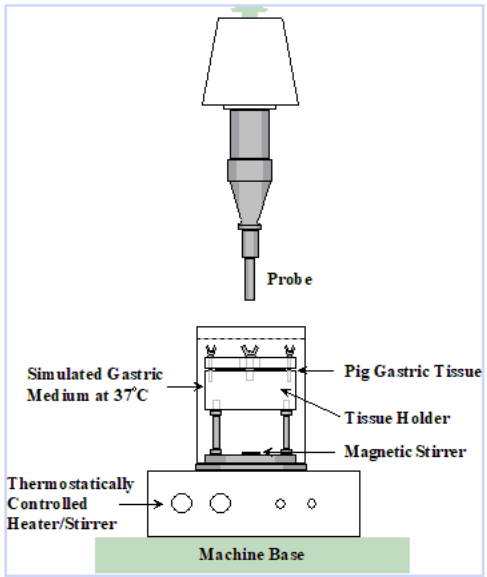


*$p<0.05$, One way ANOVA Turkey HSD *post hoc* test

Fig. 2: Comparison of peak detachment strength (tensile strength), work of mucoadhesion and viscosities 11 hydrogels containing different mucoadhesive polymers. PO = poloxamer 407 and188 only; HPMC = hydroxypropyl methyl cellulose; CMC = carboxymethyl cellulose, PEG = polyethylene glycol 6000, CAR = Carbopol 974P, POV = Povidone, HPMC+POV = 0.25% HPMC and 0.25% Povidone, HPMC+CAR = 0.25% HPMC and 0.25% Carbopol 974P

Measurement of Mucoadhesion of Tablets, Gels, Powders & Films

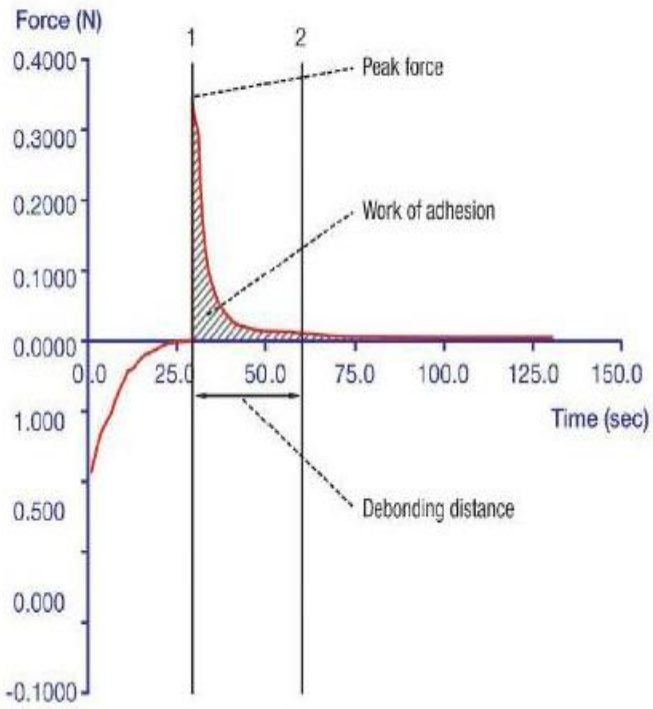
TEST SET-UP:



Typical Set-up Instructions:

Attach the cylinder probe via a probe adapter (included with the instrument) to the load cell carrier. Follow the test guidelines as described in the test method booklet.

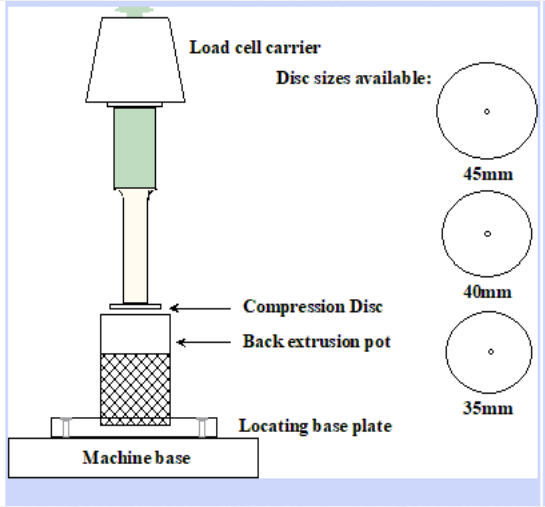
Typical plots:



1 - Mucoadhesion Test Rig
1 - Test Method Booklet
1 - 10mm Delrin Cylinder Probe

“ Improved Texture Analysis for Hydrogel Characterization: Gel Cohesiveness, Adhesiveness, and Hardness”

Julia Hurler et al .develop **a fast and reliable method** to characterize texture properties of hydrogels, namely cohesiveness, adhesiveness, and hardness



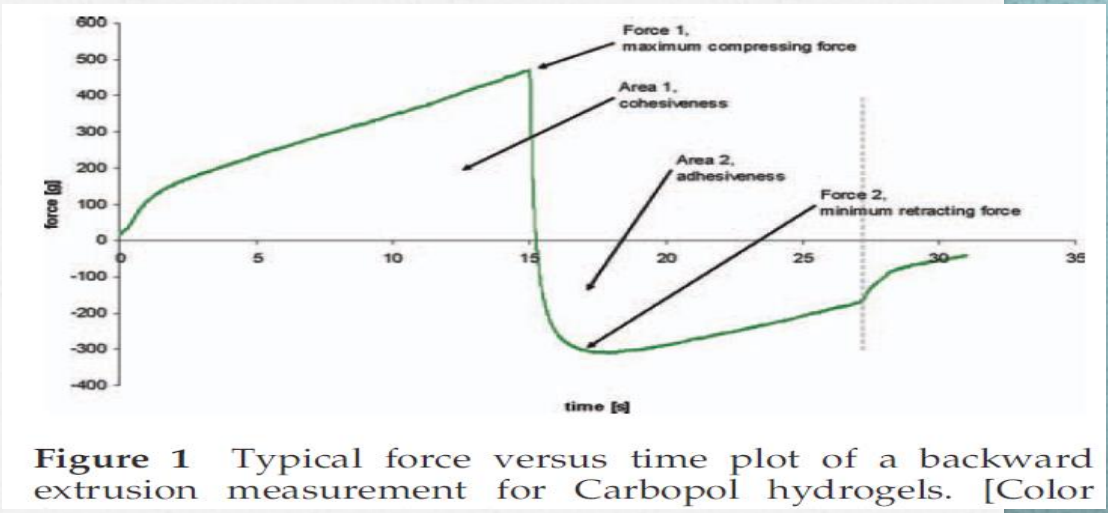


Figure 1 Typical force versus time plot of a backward extrusion measurement for Carbopol hydrogels. [Color

Texture Properties of Hydrogels Under the Optimized Measurement Conditions (n = 1)

Type of hydrogel and concentration (%; w/w)	Force 1 ± S.D. [g] (maximum compressing force; hardness)	Area 1 ± S.D. [g*s] (cohesiveness)	Force 2 ± S.D. [g] (minimum retracting force)	Area 2 ± S.D. [g*s] (adhesiveness)
Carbopol, 0.5	306.4 ± 9.7	3240.4 ± 82.0	−232.00 ± 5.9	−2676.00 ± 109.6
LMW chitosan, 5	44.6 ± 0.5	100.1 ± 0.5	−42.16 ± 0.5	-83.43 ± 0.7
Poloxamer, 22	753.2 ± 11.0	8571.6 ± 335.9	−662.25 ± 12.9	−5862.08 ± 471.5

** Journal of Applied Polymer Science, Vol. 125, 180–188 ,2012.*

**Evidence-Based Complementary and Alternative Medicine Volume 2018, Article ID 9431819*

Type of hydrogel and corresponding concentration (%; w/w)	Liposomal dispersion (%; w/w)	Force 1 ± SD (g)	Force 2 ± SD (g)
Carbopol, 0.5	0	306.4 ± 9.7	−232.0 ± 5.8
	5	293.1 ± 8.8	−229.3 ± 10.6
	10	294.4 ± 11.6	−230.8 ± 10.9
	15	286.0 ± 5.1	−221.4 ± 4.6
LMW chitosan, 6	0	170.3 ± 0.8	−123.1 ± 0.3
	5	123.0 ± 0.6	−89.1 ± 0.4
	10	97.0 ± 0.6	−75.2 ± 0.4
	15	74.7 ± 0.6	−61.8 ±0.2
MMW chitosan, 3.5	0	253.1 ± 1.1	−201.8 ± 0.6
	5	216.9 ± 0.6	−157.8 ± 0.7
	10	167.0 ± 0.7	−127.0 ± 0.5
	15	97.2 ± 0.2	−76.6 ± 0.2
HMW chitosan, 2.5	0	188.2 ± 1.0	−135.6 ± 0.6
	5	113.3 ± 1.8	−81.7 ± 0.5
	15	76.4 ± 0.7	−62.5 ± 0.1

Carbopol hydrogel retained its original texture to a great extent (93%) after the addition of liposome dispersion up to 15% (w/w)

While chitosan hydrogels with incorporated of liposomal dispersion retained only 40% of their original properties, as compared to the intact gels

“ A Quantitative Disintegration Method for Polymeric Films”

quantitative disintegration technique which can be used to characterize polymeric films in vitro without user bias

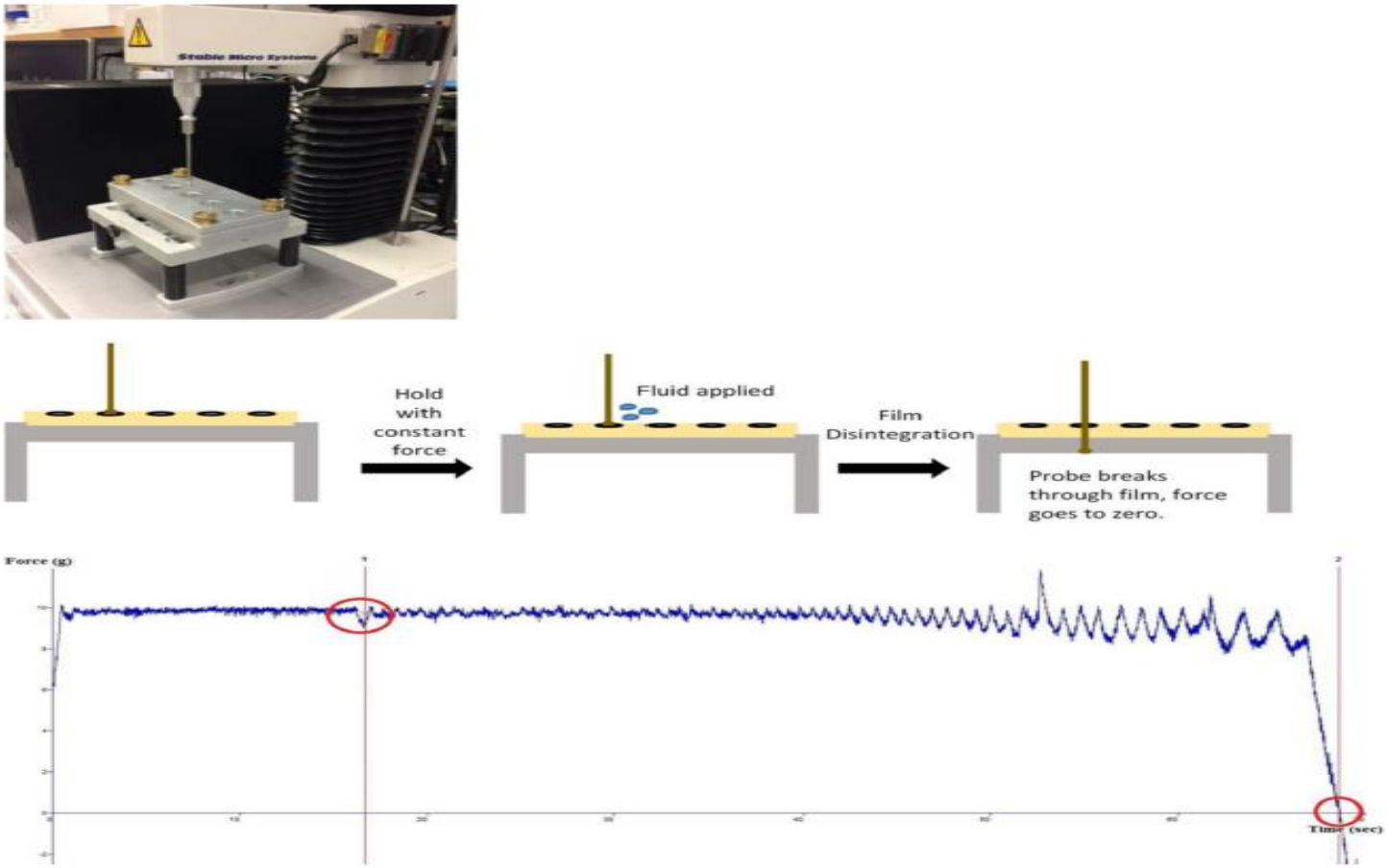


Fig. 1. Texture Analyzer Instrument Setup
(a) Instrument setup with TA-108S5 fixture and the TA-8A: 1/8" diameter rounded end ball probe. (b) Graphical schematic of setup and test positions of the Texture Analyzer disintegration technique. (c) Typical plot of force vs. time graph produced with Exponent software. Event at 15 seconds and force to zero (disintegration test end) marked in red

*J Pharm Innov. 2018 December ; 13(4): 321–329.

Disintegration Testing for VCF®

Disintegration times for VCF® (GMP product) evaluated by different users

User	Trial Number	Disintegration Time (seconds)	Standard Deviation (seconds)	% Relative Standard Deviation (RSD)
A	1	59.60	4.81	8.07
A	2	60.77	4.36	7.18
A	3	59.21	6.73	11.36
B	4	63.89	7.35	11.50
B	5	53.82	9.38	17.42
B	6	46.27	2.40	5.18
B	7	61.61	6.82	11.08

Comparison of disintegration times obtained with the visual disintegration in 1 mL of fluid for two clinically advanced vaginal films

Film	Average Disintegration Time (seconds)	Standard Deviation (seconds)	% Relative Standard Deviation (RSD)
TFV Clinical Composition	124.50	23.95	19.24
DPV Clinical Composition	227.00	40.88	18.01

Conclusion

The operation of a texture analyzer is relatively **simple**, **versatile** and **cost-effectives**

its capability to measure multiple samples within a short period of time. In addition, it is also possible to utilize the **same instrument for different dosage forms** by changing either the testing probes or the measurement parameters