Size-exclusion chromatography of xylan derivatives-the critical evaluation of macromolecular data

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6L]H H[FOXVLRQ FKURPDWRJUDSK\ RI [\ODQ

HYDOXDWLRQ RI PDFURPROHFXODU GDWD

/HHQD 3LWNOODGQPLU&KVHL\HWLDQH\HY\IDZXRFDLQHDQ7HQNDQHQ

'HSDUWPHQW RI)RRG DQG (QYLURQPHQWDO 6FLHQFHV 3 2
+HOVLQNL)LQODQG

'HSDUWPHQW RI &KHPLVWU\ 3 2 %R[8QLYHUVLW\
977 7HFKQLFDO 5HVHDUFK &HQWUH RI)LQODQG 3 2 %R[
Ž&RUUHVSRQGLQJ DXWKRU OHHQD P SLWNDQH#KHOVLQNL
YODGLPLU DVH\HY#KHOVLQNL IL FKULVWLDQH ODLQH#YWW
PDLMD WHQNDQH#KHOVLQNL IL

\$EVWUDFW

+ \GUR[\SURS\O [\ODQV ZLWK YDU\LQJ GHJUHHV RI VXEVLW
FKURPDWRJUDSK\ 0RODU PDVVHV RI WKH VDP SOHV ZHUH G
FRQYHQWLRQDO FDOLEUDWLRQ ZLWK PRODU PDVV VWDQGD
WKH FRPELQDWLRQ RI VWDWLF OLJKW VFDWWHULQJ YLVFR
7KH PRODU PDVV UHVXOWV REWDLQHG E\ WKH PXOWL GHWH
GHWHUPLQDWLRQ RI VHSDUDWH UHIUDFWLYH LQGH[LQFUHH
FROXPQ FDOLEUDWLRQ DSSURDFK ZLWK VWDQGDUG SXOOX
GLIIHUHQFHV LQ K\GURG\QDPLF YROXPHV EHWZHHQ SXOOX
PRODU PDVVHV 7KH GHJUHH RI K\GUR[\SURS\ODWLRQ DIIH
RI WKH SRO\PHU FKDLQV 0DUN +RXZLQN SDUDPHWHUV DQG
K\GUR[\SURS\O VXEVLWXHQWV UHGXFH WKH IOH[LELOLW\
PRUH H[WHQG

.H\ZRUPDQV K\GUR[\SURS\ODWLRQ VL]H H[FOXVLRQ FKUR

,QWURGXFWRQ

,QWHUHVW LQ WKH XWLOL]DWLRQ RI ELRPDVV KDV LQFUHDV
7ZR JOREDO LVVXHV KDYH UDLVHG LQWHUHVW LQ ILQGLQJ
QHFHVVLW\ RI XVLQJ UHQHZDEOH FDUERQ VRXUFHV LQVWH
JUHHQKRXXVH JDV HPLVLRQV 7KH XVH RI ELRSRO\PHUV H[V
V\QWKHWLF SRO\PHUV LV RQH LPSRUWDQW ZD\ RI XWLOL]LO
WKHLU QDWLYH IRUPV WKH SURSHUWLHV RI ELRSRO\PHUV
HQ]\PDWLF RI WKH SRO\PHULF FKDLQV 7KLV VWXG\ IRFXVH
FKHPLFDOO\ PRGLLHG ELUFK [\ODQ ;\ODQV WKH FHOO ZD
JURXS RI KHPLFHOOXORVHV DQG FRQVWLWXWH WKH VHFRQ
FHOOXORVH > @

7KH FRPSRVLWLRQ RI [\ODQV YDULHV GHSHQGLQJ RQ WKH S
FRQWDLQV JOYFXURQR[\ODQV *; ZKLFK LQ WKHLU QDWLY
[\ORS\UDQR;VOEDFNERQH ZLWK : OLOOLHRS2UDQR\O XU
DFLG UHVLGXHV 0H*OFSS\$ DQG DFHW\O JURXSV DWWDFKHG
H[WUDFWLRQ WKDW LV PDLQO\ XVHG IRU KHPLFHOOXORVHV
UHPRYLQJ WKH DFHW\O JURXSV 'HDFHW\ODWLRQ GHFUHDV
NUDIW SXOS IXUWKHU PRGLLHV WKH [\ODQ VWUXFWXUH E\
IURP WKH NUDIW SXOS SURFHVV HPSOR\HG LQ WKLV VWXG
RI DSSUR[LPDWHO\ PHT J GU\ ZHLJKW > @ %\FKHPLFD
K\GUR[\SURS\ODWLRQ RI DONDOL H[WUDFWHG [\ODQV WKH
VWXGLHV RQ K\GUR[\SURS\ODWLRQ RI [\ODQV XVLQJ HLWK
UHDJHQWV KDYH VKRZQ WKH SRWHQWLDO RI WKLV FKHPLF
+\GUR[\SURS\O [\ODQ +3; KDV EHHQ VKRZQ WR EH D SRWH

FRXOG EH XVHG LQ ELRGHJUDGDEOH SDFNDJLQJ > @
K\GUR[\DON\ODWLRQ L H LQWURGXFWRQ RI K\GUR[\HWK\
& DQG RU & LQ RUGHU WR LPSURYH LWV ZDWHU VROXELO
WKLFNHQHU DQG HPXOVLQRQ VWDELOL]HU LQ WKH IRRG LQG
SKDUPDFHXWLFDO LQG XVWU\ DV D ELQGLQJ DJHQW LQ WDE
@

ORODU PDVV DQG HVSHFLDOO\ PRODU PDVV GLVWULEXWLR
LQIOXHQLQJ WKH SRO\PHU SURSHUWLHV > @ ,Q RUGHU W
WKH SRO\PHULF VDPSON QHHGV WR EH VHSDUDWHG XVLQJ
6(& RU ILHOG IORZ IUDFWLRQDWLRQ))) 7KH VLPSONHV
GLVWULEXWLRQ LV WR FRXSON WKH VHSDUDWLRQ WHFKQLT
FRPPRQO\ GLIIHUHQWLDO UHIUDFWLYH LQGH['5, RU XOWU
DQDO\VLV RI ORZ GLVSHUVLW\ PRODU PDVV VWDQGDUGV XV
VDPSONHV 7KH GHWHFWRU VLJQDO LQWHQVLW\ WKXV JLYHV
HOXWLRQ YROXPH GHILQHV WKH PRODU PDVV ZKHQ WKH U
WKH HOXWLRQ YROXPHV RI WKH VWDQGDUGV 7KLV VR FDO
SURQH WR HUURUV FDXVHG E\ WKH GLIIHUHQW PRODU PDVV
WKH VWDQGDUGV XVHG IRU FDOLEUDWLRQ \$OVR PRODU P
VHQVLWLYH WR HUURUV FDXVHG E\ HQWKDOSLF LQWHUDFW
\$GGLQJ RWKHU GHWHFWRUV WR WKH GHWHFWRU WUDLQ HY
VFDWWHULQJ GHWHFWRUV >0\$/6@ DQG G\QDPLF OLJKW VFD
IRU WKH GHWHUPLQDWLRQ RI 'DEVROXWH' PRODU PDVVHV
HDFK VHSDUDWHG IUDFWLRQ > @

\$V DOUHDG\ GLVFXVVHG LQWHUHVW LQ PRGLILFDWLRQ RI
DOVR EH JURZLQJ GHPDQG IRU WKH GHYHORSPHQW RI DQDO
ELRPDFURPROHFXOHV (YHQ WKRXJK WKH DQDO\WLFDO WRF
DOUHDG\ H[LVWV FULWLFDO HYDOXDWLRQ RI WKH XVH RI V
FRPSOH[DQG KHWHURJHQHRXV VWUXFWXUHV LV LPSRUWDQ
YDU\LQJ GHJUHHV RI K\GUR[\SURS\ODWLRQ ZHUH FKDUDFW
OLJKW VFDWWHULQJ YLVFRPHWHU DQG GLIIHUHQWLDO UH
FDOLEUDWLRQ HPSOR\LQJ QDUURZ GLVSHUVLW\ SXOOXODQ
7KH DFFXUDF\ RI ERWK WHFKQLTXHV ZDV HYDOXDWHG IRU
FKHPLFDO FRPSRVLWLRQ RI WKH +3; VDP SOHV ZDV WDNHQ
GHHFWLRQ GDWD ZDV HYDOXDWHG 7KL V ZDV DFFRPSOLV
K\GUR[\SURS\ODWLRQ GHJUHH RQ WKH UHIUDFWLYH LQGH[
ERWK IRU FRQFHQWUDWLRQ GHHWUPLQDWLRQ E\ GLIIHUHQ
PRODU PDVV E\ OLJKW VFDWWHULQJ

0DWHULDOV DQG PHWKRGV

0DWHULDOV

7KH K\GUR[\SURS\O [\ODQV XVHG KHUH ZHUH SURGXFHG SU
DONDOLQH H[WUDFWHG ELUFK [\ODQ ZDV K\GUR[\SURS\ODW
FKHPLFDOO\ PRGLILHG [\ODQV IRUPHG GLVSHUVLRQV ZLWK
3DUW RI WKH GLVSHUVLRQ ZDV IUHH]H GULHG IRU 6(& D
'062 IRU WKH 6(& ZDV REWDLQHG IURP)LVKHU 6FLHQWLIL
EURPLGH /L%U ZDV REWDLQHG IURP 6LJPD \$OGULFK 6WHI

SXOOXODQ VWDQGDUGV FDPH IURP \$JLOHQW 3RO\PHU /DER
\$QDO\WLFV /DQGVEHUJ /HFK *HUPDQ\

6(& DQDO\VLV

7KH 6(& LQVWUXPHQW FRQVLVWHG RI DQ LQWHJUDWHG DXW
ODOYHUQ ,QVWUXPHQWV /WG :RUFHVWHUVKLUH 8. D FRO
6DLQWH)R\ OD *UDQGH)UDQFH D FRPELQHG VWDWLF OL
9LVFRWHN 'XDO 'HWHFWRU ODOYHUQ ,QVWUXPHQWV /W
GHWHFWRU 9LVFRWHN 9(ODOYHUQ ,QVWUXPHQWV /WG
LQFOXGHG WZR VFDWWHULQJ DQJOHV *f* DQG *f* 7KH VHSI
-RUGL -RUGL /DEV ODQVILHOG 86\$ FROXPQV ; VWUHDP P
J PRO WR J PRO DQG ; VWUHDP cu GLPHQVLRQ
R& 7KH IORZ UDWL ZDV PO PLQ DQG WKH LQMHFWRQ \
GLVVROYHG LQ PRELOH SKDVH '062 FRQWDLQLQJ 0 /L%U
VDPSOHV ZHUH ILOWHUHG XVLQJ —P V\ULQJH ILOWHUV
EHIRUH DQDO\VLV 7KH PRODU PDVV DYHUDJHV ZHUH FDOFX
PHWKRG > @ RU E\ FRQVWUXFWLQJ D UHODWLYH FDOLE
VWDQGDUGV SXOOXODQV ZLWK QRPLQDO PRODU PDVVHV R
J PRO &DOLEUDWLRQ FXUYH LV VKRZQ DV 6XSSOHPH
YDOXHV ZHUH GHWHUPLQHG EDVHG RQ WKH '5, VLJQDOV

6SHFLILF UHIUDFWLYH LQGH[LQFUHPHQW ~Q ~F GHWHUPLQ

6SHFLILF UHIUDFWLYH LQGH[LQFUHPHQW ~Q ~F YDOXHV IR
>'6@ RI DQG ZHUH GHWHUPLQHG RIIOLOQH XVLQJ
ZDYHOHQJWK RI QPJKW DWW 7HFKQRORJ\ &R 3;6VDP SOHV DZBD
GLVVROYHG LQ '062 FRQWDLQLQJ 0 RI /L%U DW VL[FRQ
DQG PJ PO DQG HDFK VROXWLRQ ZDV LQMHFWHG GLUHFW
PO V\ULQJHV 7KH WHPSHUDWXUH RI WKH UHIUDFWRPHWHU
7HFKQRORJ\ &R ZDV XVHG IRU GDWD DFFRPSWHFWLRQ DQG
WKH VORSHV RI D SORW RI FRQFHQWUDWLRQ YHUVXV GLIHF

5HVXOWV DQG GLVFXVVLRQ

7KH VHULHV RI +3; VDP SOHV ZLWK YDU\LQJ '6 ZHUH SURGX
XVLQJ SURS\OHQH R[LGH DV D UH DJHQW IRU GHULYDWL]DW
TXDQWLWDWLYH SHFWURVFRS\ DQG DYHUDJH '6 YDOXHV RI
7KH +3; VDP SOHV ZHUH VHSDUDWHG DQG FKDUDFWHUL]HG X
SXOOXODQ FDOLEUDWLRQ VLQJOH FRQFHQWUDWLRQ VHQV
GHWHFWRUV OLJKW VFDWWHULQJ >/6@ YLVFRPHWU\ >9,6&
RUJDQLF VROYHQW ZDV XVHG DV WKH HOXHQW '062 0
DTXHRXV HOXHQW ZDV DOVR WHVWHG IRU +3; VDP SOHV EX
UHYHDOHG WKDW +3; FKDLQV ZHUH QRW GLVVROYHG DW WK
VDP SOHV FRQWDLQHG KLJK PRODU PDVV DJJUHJDWHV SUH
VKRZQ WKDW WKH PRODU PDVVHV RI QLWUHQ H[WUDFWHG E

a J PRO > @ \$TXHRXV HOXHQW ZDV DQ REYLRXV ILUV
SXUSRVH RI K\GUR[\SURS\ODWLRQ LV WR LQFUHDVH WKH ZD
FHOOXORVH DQG YDULRXV KHPLFHOOXORVHV 7KH DONDOL
PDWHULDO IRU K\GUR[\SURS\ODWLRQ ZDV QRW VROXEOH LG
IRU 6(& DQDO\VHV RUJDQLF RU DTXHRXV

)LJ 6(& FKURPDWRJUDPV IRU K\GUR[\SURS\O [\ODQV ZLWK Y
7KH YLVFRPHWURF HDEKQ\ODQ LV SORWWHG DFURVV WKH S
GHHHFWRU

ORODU PDVV GHWHUPLQDWLRQ RI K\GUR[\SURS\O [\ODQV E\

7KH 6(& FKURPDWRJUDPV RI +3; VDP SOHV GHWHFWHG E\ '5,
RQO\ PLQRU GLIIHUHQFHV LQ WKH UHWHQWLRQ YROXPHV F
VWDUWLQJ PDWHULDO IRU K\GUR[\SURS\ODWLRQ ZDV WKH V
LQ WKH PRODU PDVVHV VKRXOG KDYH UHVXOWHG IURP WKH
7KH SHUFHQW DJH LQFUHDVH LQ PRODU PDVV FDQ EH FDOFX
NQRZQ KHUH WKH '6 LV REWDLQHG IURP 105 VSHFWURVFR